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147547

FINAL REPORT

THE DEVELOPMENT OF SPACECRAFT EXPLOSIVES FOR THE LYNDON B. JOHNSON SPACE CENTER

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(NSWC-841/NASA)

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THE DEVELOPMENT OF SPACECRAFT EXPLOSIVES FOR THE LYNDON B. JOHNSON SPACE CENTER

1. INTRODUCTION

1.1 This is the Naval Surface Weapons Center (NAVSURFWPNCEN) Final Report on work done on the "Space Shuttle Explosives" task for the National Aeronautics and Space Agency (NASA), Lyndon B. Johnson Space Center at Houston, Texas. This work carried out for NASA under task NSWC-841/NASA completes the planned effort in Phase II of the NASA work statement. Phase I work was reported in the 1974 Annual Progress Report. The purpose of the work was to characterize several heat resistant secondary explosives suitable for use in spacecraft hardware) and select a prime candidate for further study. The design goal criterion was "no significant degradation of the explosive after exposure to 315°C (600°F).

1.2 The final selection of a prime candidate explosive for further study was made from three candidates, PYX (2,6-bis (picrylamino) -3,5-dinitropyridine)[1,2] ONT (octanitro-m-terphenyl)[3,4] and TPT (2,4,6 Tripicryl-s-triazine)[5] following performance tests in detonating cords. TPT was the final selection and was further characterized and evaluated in order to obtain data for a procurement specification.

2. EXPOSURE OF PYX DETONATING CORDS TO ELEVATED TEMPERATURES

2.1 PYX, one of the three heat resistant explosives being investigated, was procured from the Los Alamos Scientific Laboratory (LASL). Scanning Electron Microscope (SEM) photomicrographs of this PYX (ID 1817) are shown in Figure 1. The literature publication[1] on PYX reveals the product was washed with concentrated nitric acid, water, and methanol, and then dried at 140°C. There were no published vacuum thermal stability results for the laboratory or pilot plant synthesized explosive. Test results[2] from NAVSURFWPNCEN show a large quantity of gas for the 20 minute surge (about three times the acceptable maximum amount) for the vacuum thermal stability. In

(1) Coburn, M. D. and Singleton, J. L., J. Heter. Chem., 9, 1039, 1972
(2) Kilmer, E., "A Characterization Study of Several Heat Resistant Explosives," NOLTR 74-177, 4 Oct 1974
(3) Dacons, J. C., "Heat Resistant Explosives XXIII. The Preparation and Properties of 2,2",4,4',4",6,6',6"-- Octanitro-m-terphenyl, ONT," NOLTR 66-179, 5 Oct 1966
(4) Dacons, J. C., US Patent #3,592,860, "Octanitroterphenyl," 13 Jul 1971 patented
(5) Dacons, J. C., US Patent #3,755,321, "2,4,6-tripicryl-s-triazine," 28 Aug 1973 patented

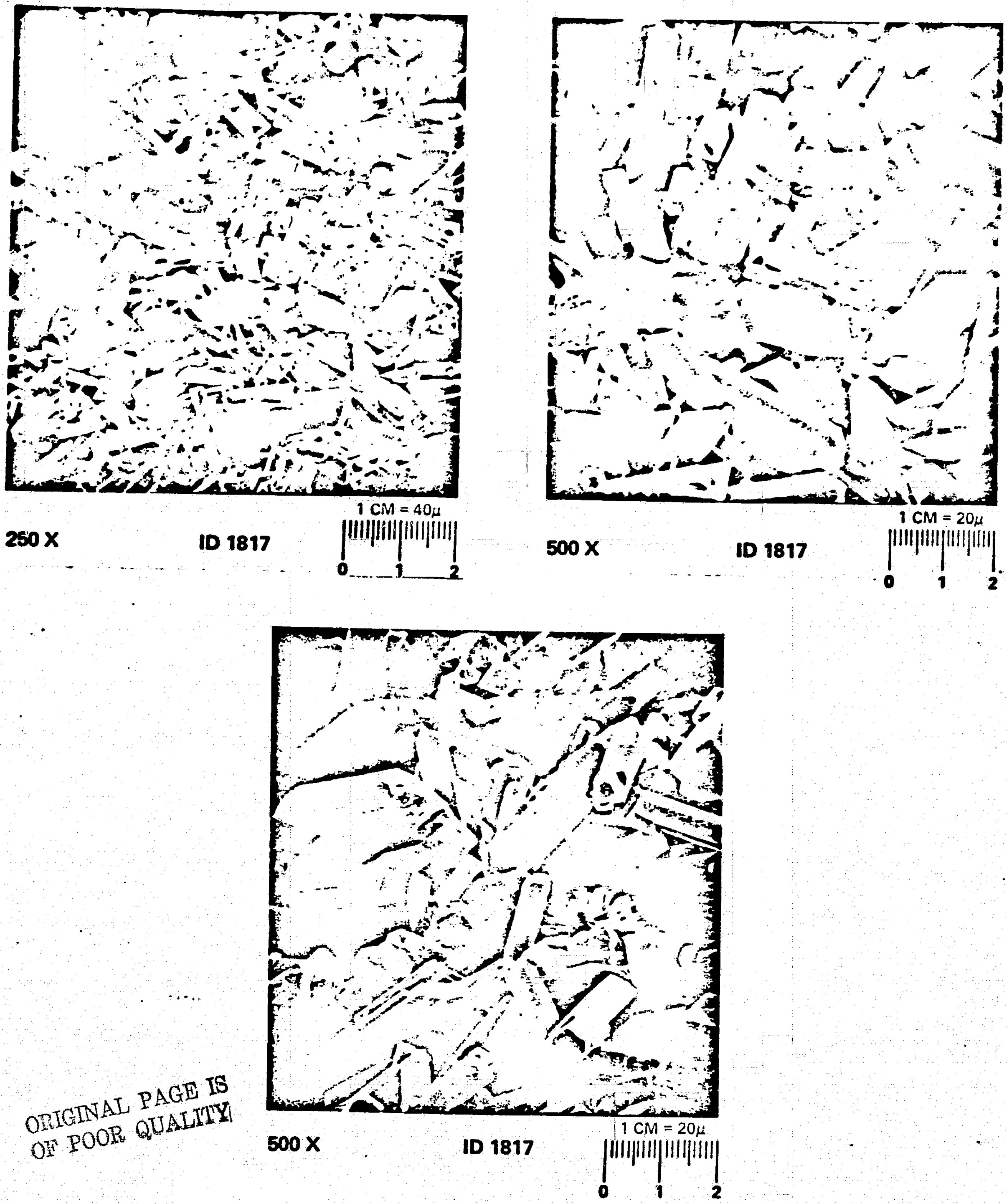

FIG. 1 SEM PHOTOMICROGRAPHS OF PYX (ID1817) "AS REC'D" FROM LASL

view of these results, LASL (Dr. M. Coburn) was contacted by NAVSURFWPNCEN (Dr. J. Dacons) by telephone. Dr. Coburn recommended that the explosive received from the LASL pilot plant be "cleaned-up" by dissolving the PYX in red fuming nitric acid and recrystallizing it. This was done and the PYX was resubmitted for vacuum stability testing. The surge value was reduced to an acceptable quantity. The recrystallized PYX (ID 1854) is shown in Figure 2.

2.2 A quantity of PYX was furnished as GFE to the Ensign Bickford Company to be fabricated into detonating cords. Cords were loaded at 0.4-0.6 g/m (2-3 gr/ft) and 2-3 g/m (10-15 gr/ft) in aluminum and silver sheaths. These cords were sealed into copper tubes by cold welding and were then exposed to elevated temperatures of 300°C, 315°C, and 326°C. After exposure, they were removed and test fired at room temperature. The detonation velocities of the cords are shown as a function of the exposure time in Figures 3, 4, 5, and 6. Each point on the curve represents a single sample test firing.

2.3 From the results of tests after subjecting PYX to 300°C for 5 to 8 hours, it is doubtful that this material will be useful after long periods of time at elevated temperatures. All samples of aluminum and silver sheathed detonating cords failed to support detonation after 5 to 8 hours at 300°C.

2.4 When the temperature was increased to 315°C for a shorter time (1/2, 1 and 2 hours), the performance was not degraded as much as at 300°C for the longer times. Refer to Figures 3, 4, 5, and 6. A further penalization of the explosive was done by raising the temperature to 326°C. Here the degree of degradation appears to be related to the core load. The 0.4 g/m (2 gr/ft) cords in both aluminum and silver performed very badly at 315°C. However, even with the larger core load, performance is questionable after one hour exposure to 326°C.

3. EXPOSURE OF ONT DETONATING CORDS TO ELEVATED TEMPERATURES

3.1 ONT, one of the heat resistant explosives of the group of materials to be investigated for NASA, was fabricated into detonating cords and subjected to elevated temperatures. ONT was synthesized in 1966 by Dacons of this Center[3,4]. Some of the first work on studying the performance in detonating cords was done by Kilmer of this Center[5] in 1970 under the NASA "High and Low Temperature Resistant Devices Task."

3.2 As a part of the present task, it was decided (because of ONT's good thermal properties) to investigate ONT at higher temperatures than considered in the past. ONT loaded (0.4 g/m and 2 g/m) aluminum and silver sheathed cords were sealed into copper tubes by cold welding. They were exposed to elevated temperatures of 280°C, 300°C, and 315°C in the same way as were the PYX cords. SEM photomicrographs of the ONT explosive are shown in Figure 7.

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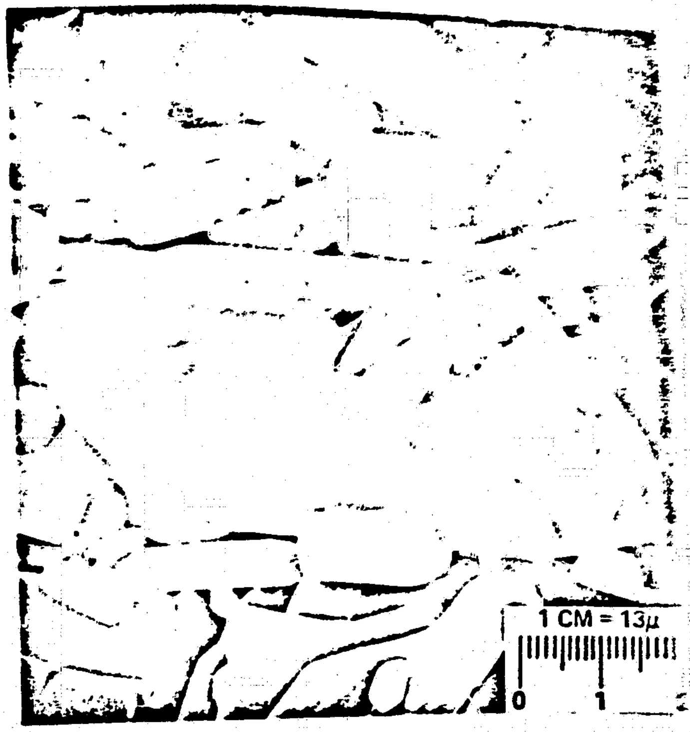

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FIG. 2 SEM PHOTOMICROGRAPHS OF PYX (ID1854) RECRYSTALLIZED FROM RED FUMING NITRIC ACI

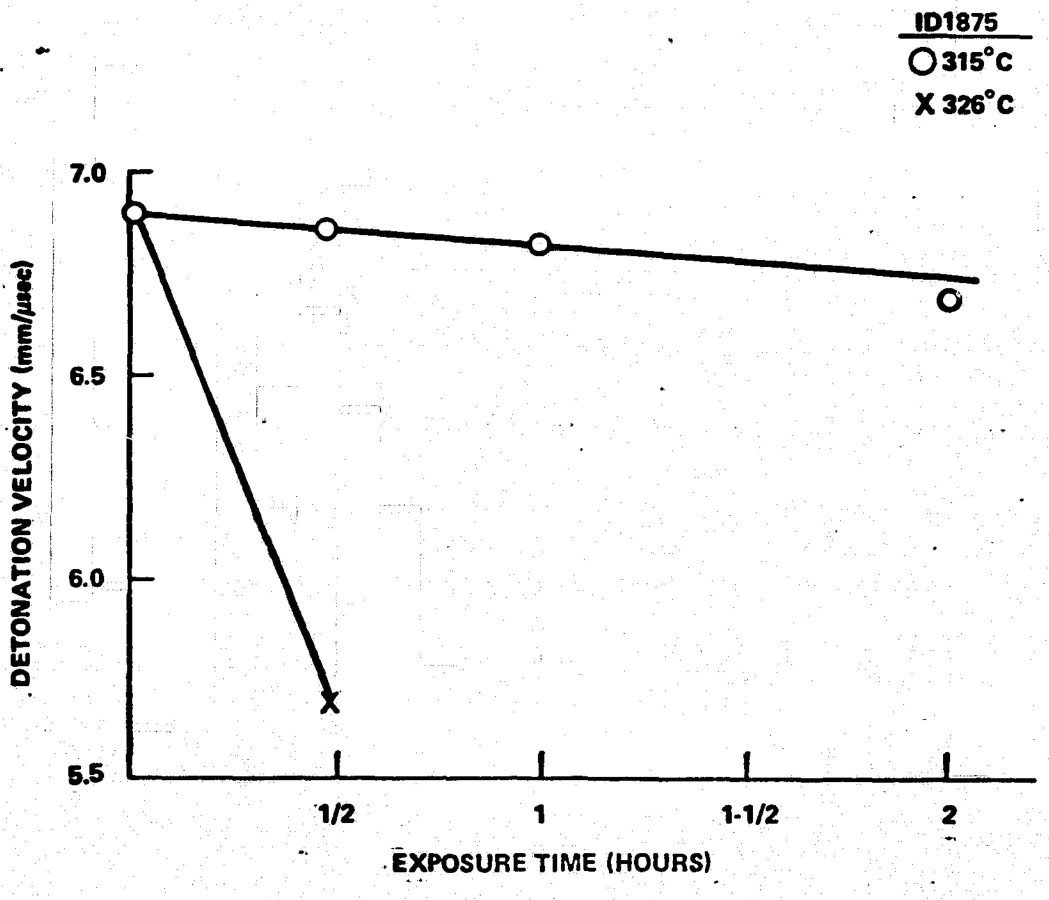


FIG. 3 THE DETONATION VELOCITY OF PYX IN ALUMINUM MDC (0.5g/m OR 2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

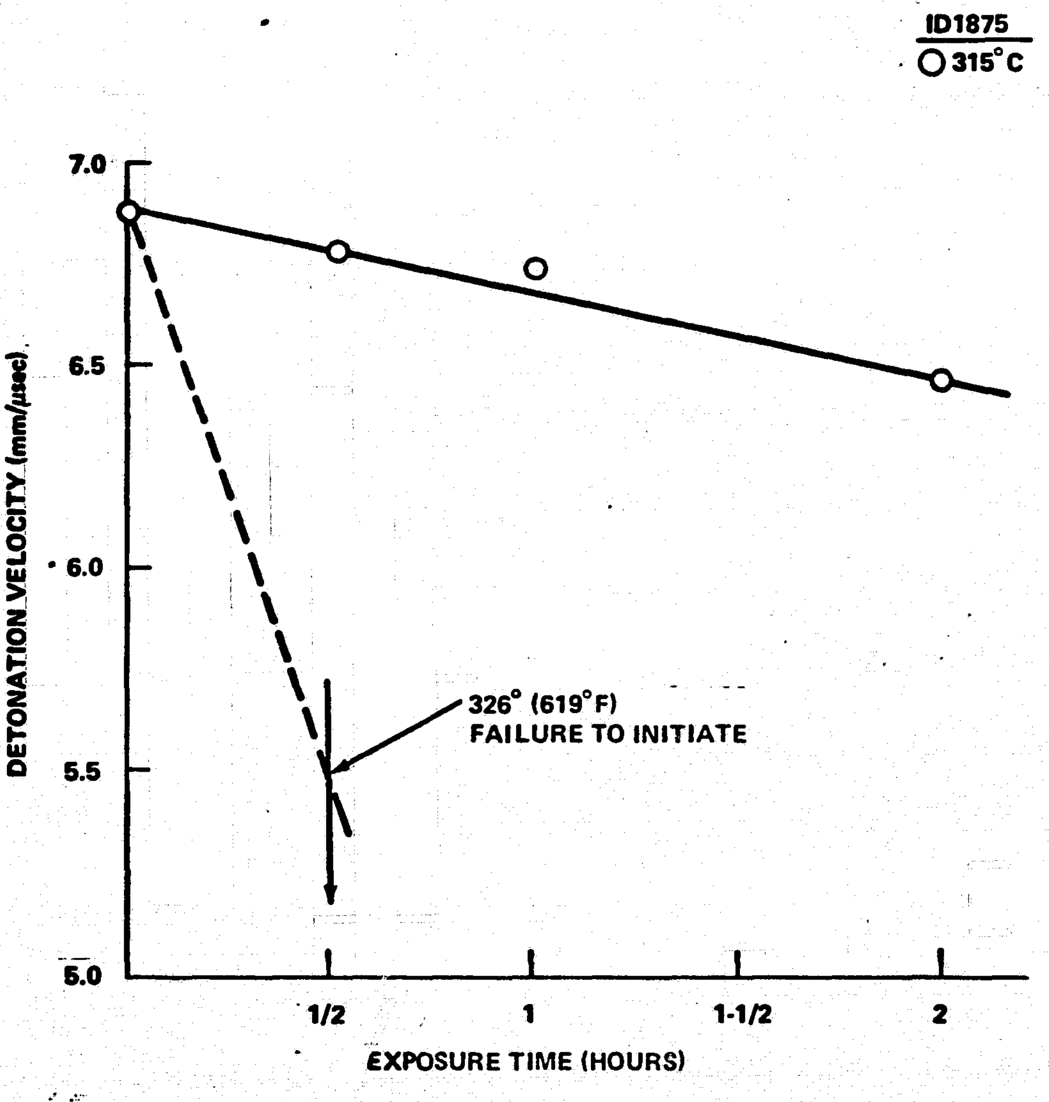


FIG. 4 THE DETONATION VELOCITY OF PYX IN SILVER MDC (0.5g/m OR 2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

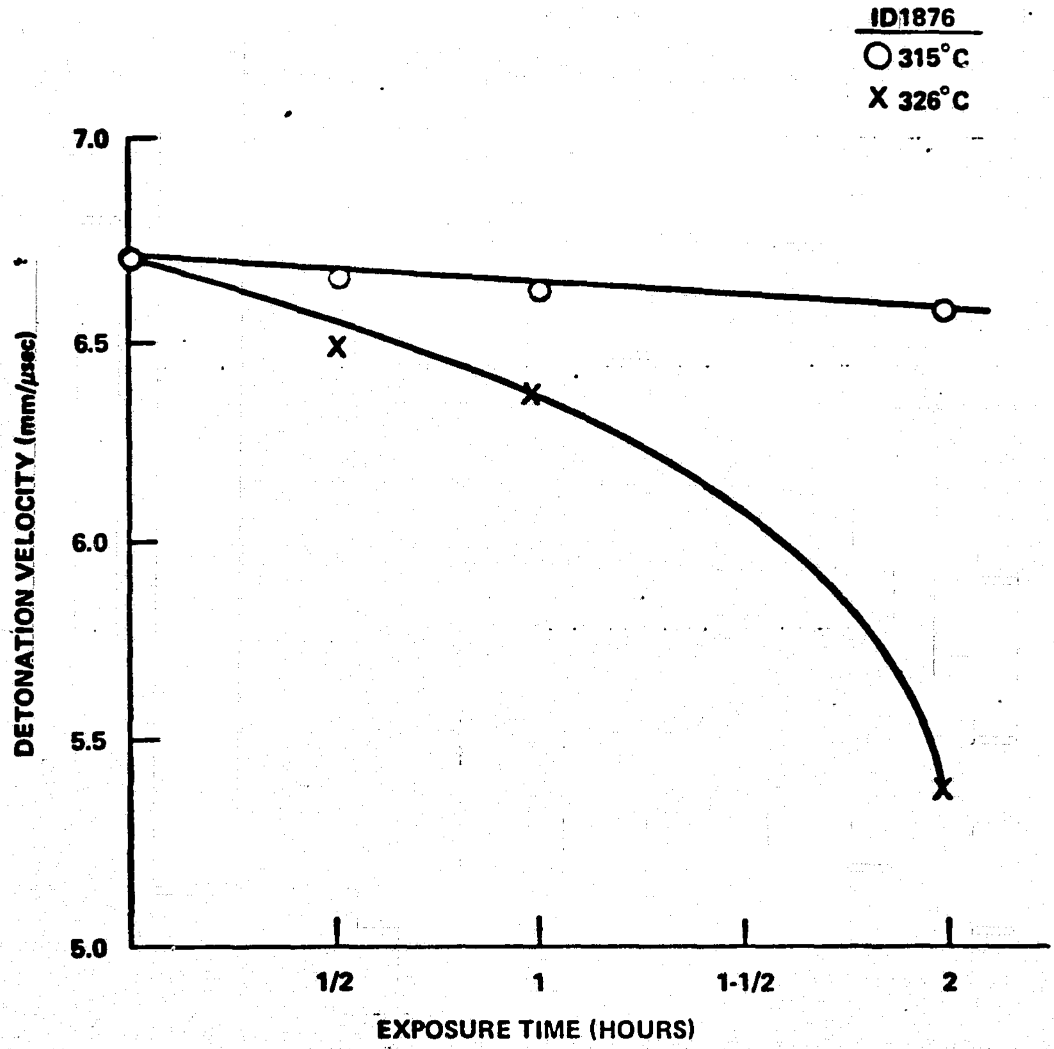


FIG. 5 THE DETONATION VELOCITY OF PYX IN ALUMINUM MDC (2.4g/m OR 12gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

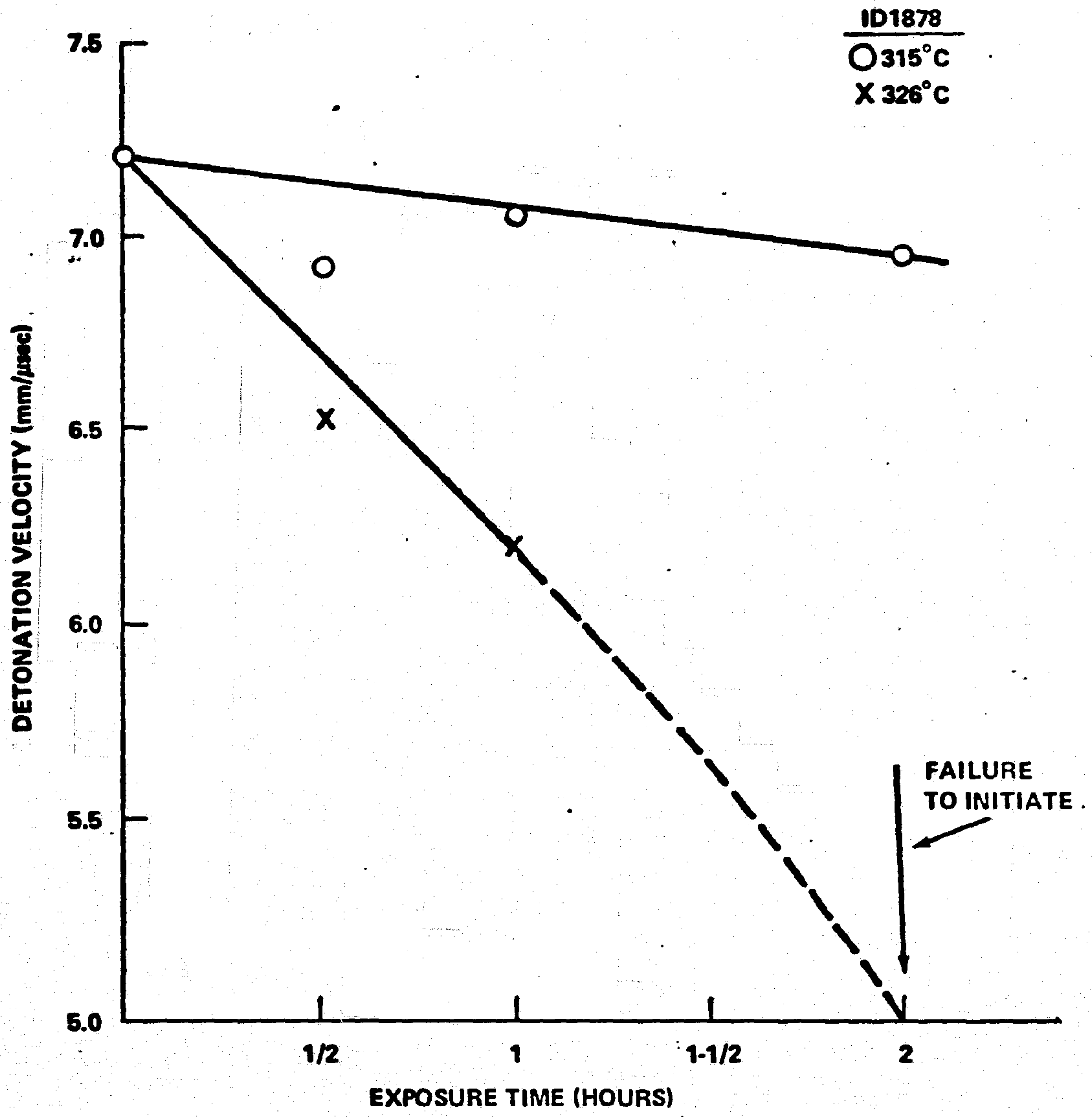


FIG. 6 THE DETONATION VELOCITY OF PYX IN SILVER MDC(2g/m OR 10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

50 X ID 1803 1 CM = 200μ 0 1 2

100 X ID 1803 1 CM = 100μ 0 1 2

250 X ID 1803 1 CM = 40μ 0 1 2

FIG. 7 SEM PHOTOMICROGRAPHS OF ONT (ID1803)

3.3 Cords containing ONT were fired at room temperature after exposure to elevated temperatures. The detonation velocities of the cords are shown as a function of exposure time at elevated temperatures in Figures 8 through 19. It should be noted that the characterization studies on ONT indicate considerable gassing at 300°C in the vacuum thermal stability test. Therefore, it was expected that performance would probably be limited to temperatures below 300°C.

3.4 From the results of the tests, the following observations were made:

(a) At the outset of the exposure to elevated temperature, i.e., the first one-half to one hour, the aluminum cords decrease a few hundred meters/sec in detonation velocity relative to the "as received" cords, but the change in velocity decreases with increased exposure time over a 7-1/2 hour period at 280°C and 300°C. This loss of detonation velocity is typical of other heat resistant explosives, such as HNS, when fabricated into cords. However, the change in detonation velocity is significant at 315°C (600°F) (see Figures 12 and 13), and the ONT is degrading fast enough so as to make its use questionable in hardware at this temperature.

(b) The silver cords do not show the reduction of detonation velocity at the outset of the high temperature exposure except at the 315°C temperature. The 0.4 g/m (2 gr/ft) material shows the typical degradation after one-half hour at 315°C, but the 2 g/m (10 gr/ft) material does not begin to degrade until after one hour of exposure. The 315°C exposure appears to be too high for reliable performance.

4. EXPOSURE OF TPT DETONATING CORDS TO ELEVATED TEMPERATURES

4.1. TPT was fabricated into detonating cords. The core loadings were 0.4-0.6 g/m (2-3 gr/ft) and 2-3 g/m (10-15 gr/ft) in aluminum and silver sheaths. Lengths of detonating cords (10.2 cm or 4.0 inches), containing TPT, were sealed into copper tubes by cold welding the ends of the tubes together, exposed to elevated temperatures of 300°C, 315°C, and 324°C, removed, and fired at room temperature. The detonation velocities of these cords are shown as a function of the exposure time in Figures 20, 21, 22, and 23.

4.2 At 315°C (600°F), only the 2 g/m TPT in aluminum showed no degradation. The other cords showed reduced exposure. Figure 21 reveals very little degradation after two hours at 315°C (597°F) for the 2 g/m TPT in aluminum. As a penalization of the design goal of 315°C/1 hour, and to further characterize the 10 gr/ft TPT, samples were tested after one-half hour at 326°C (619°F). Some degradation of velocity occurred.

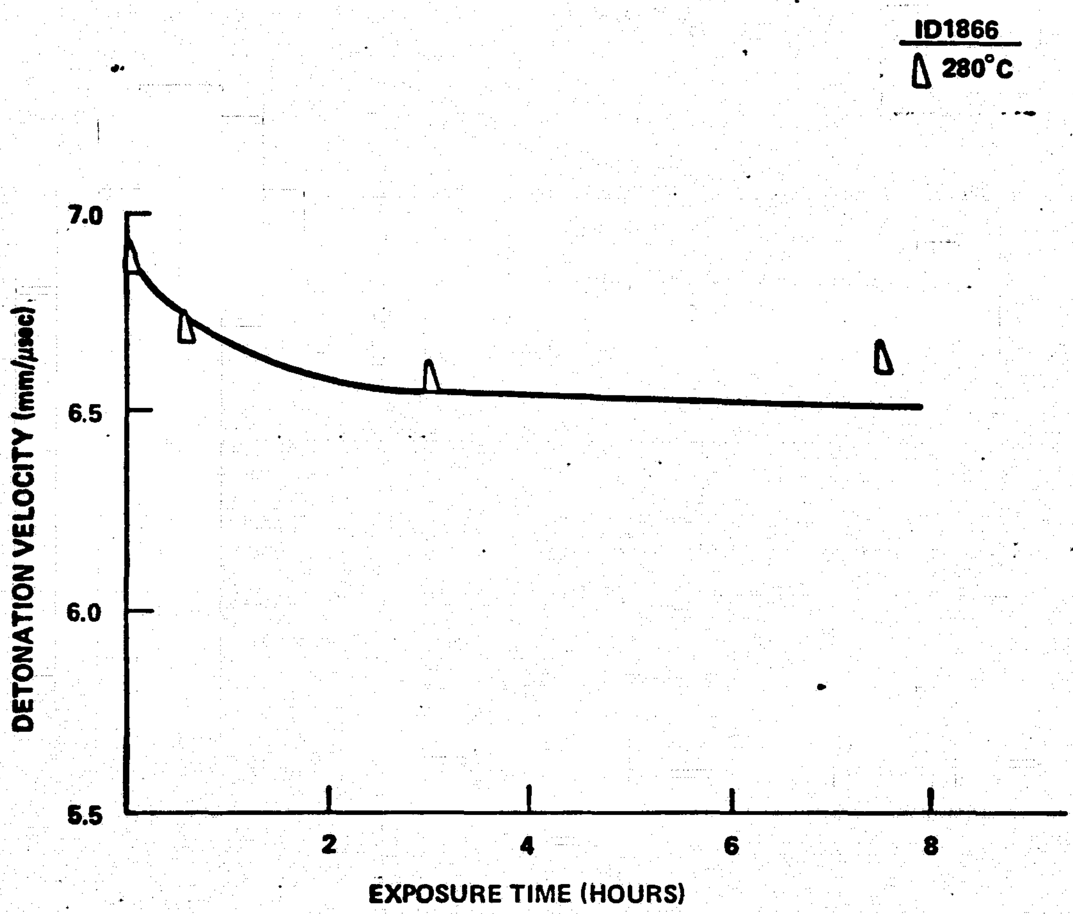


FIG. 8 THE DETONATION VELOCITY OF ONT IN ALUMINUM MDC (0.5g/m OR 2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

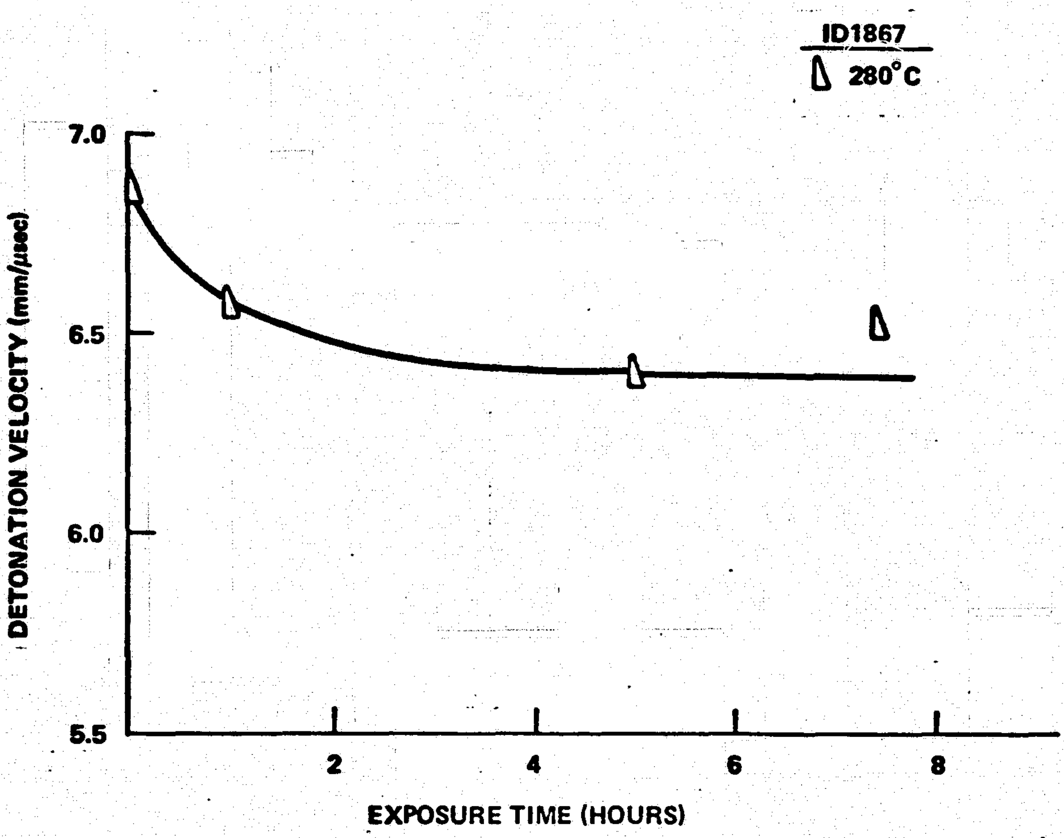


FIG. 9 THE DETONATION VELOCITY OF ONT IN ALUMINUM MDC (2g/m OR 10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

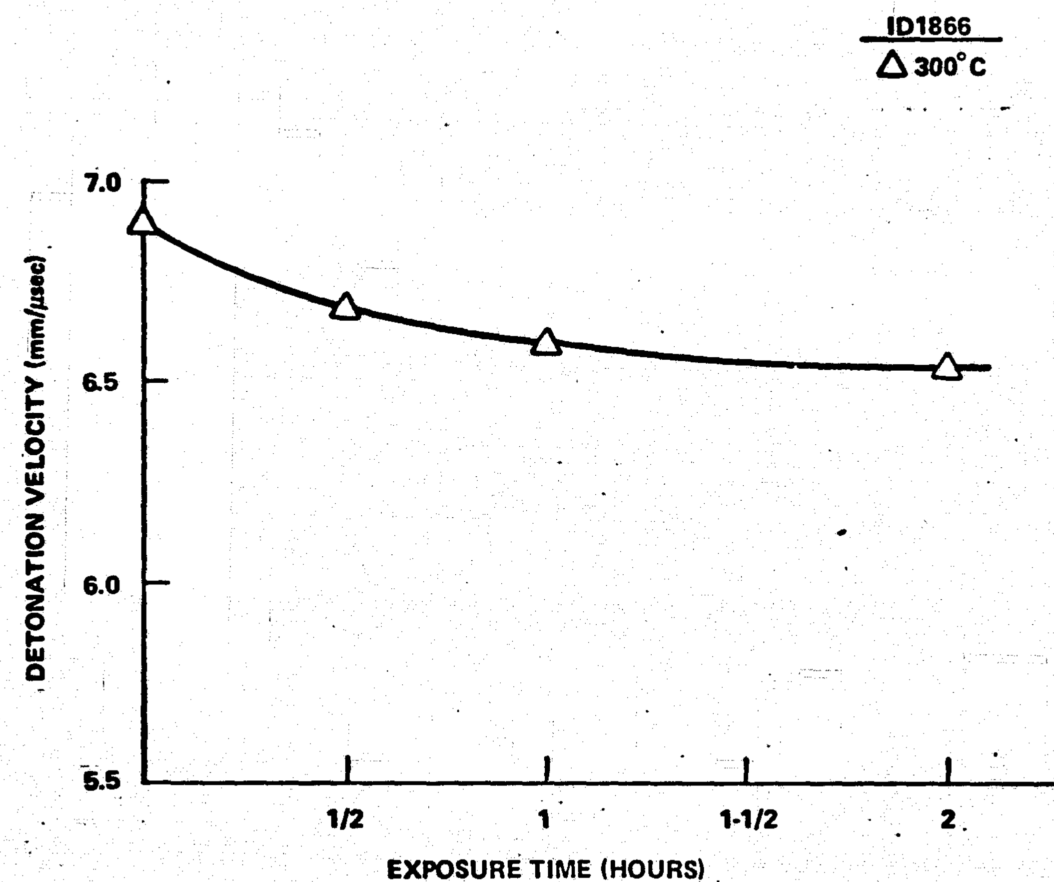


FIG. 10 THE DETONATION VELOCITY OF ONT IN ALUMINUM MDC (0.5g/m OR 2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

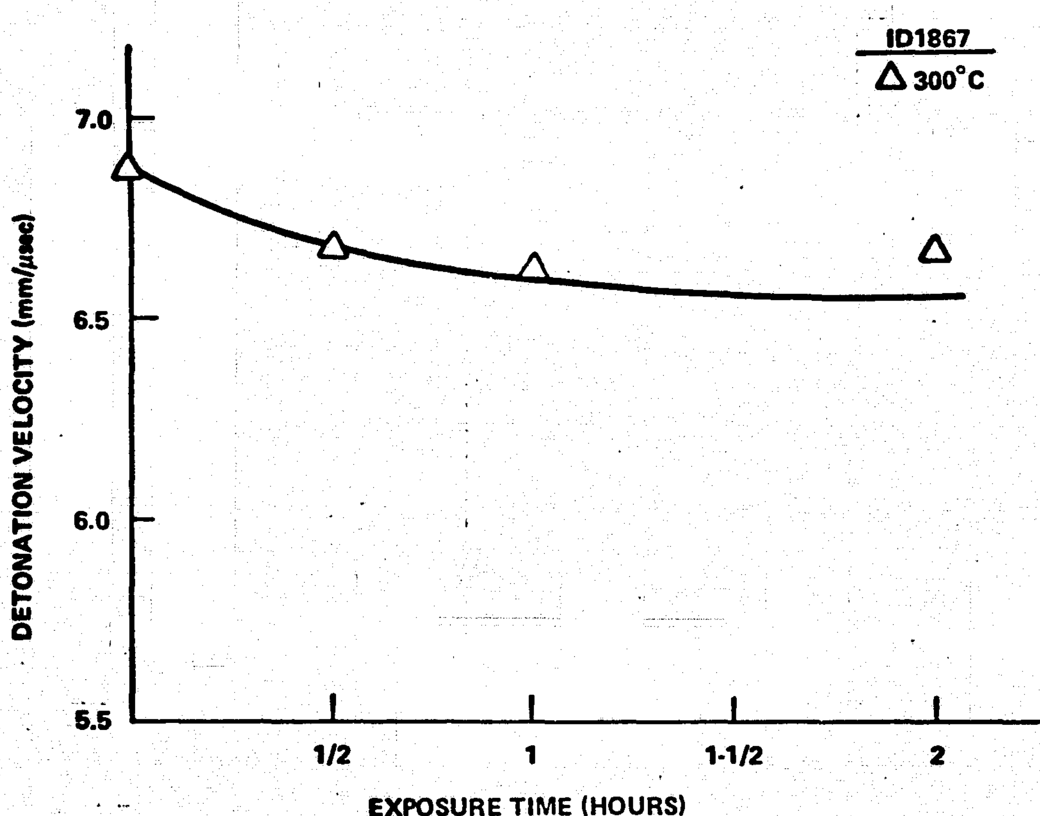


FIG. 11 THE DETONATION VELOCITY OF ONT IN ALUMINUM MDC (2g/m OR 10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

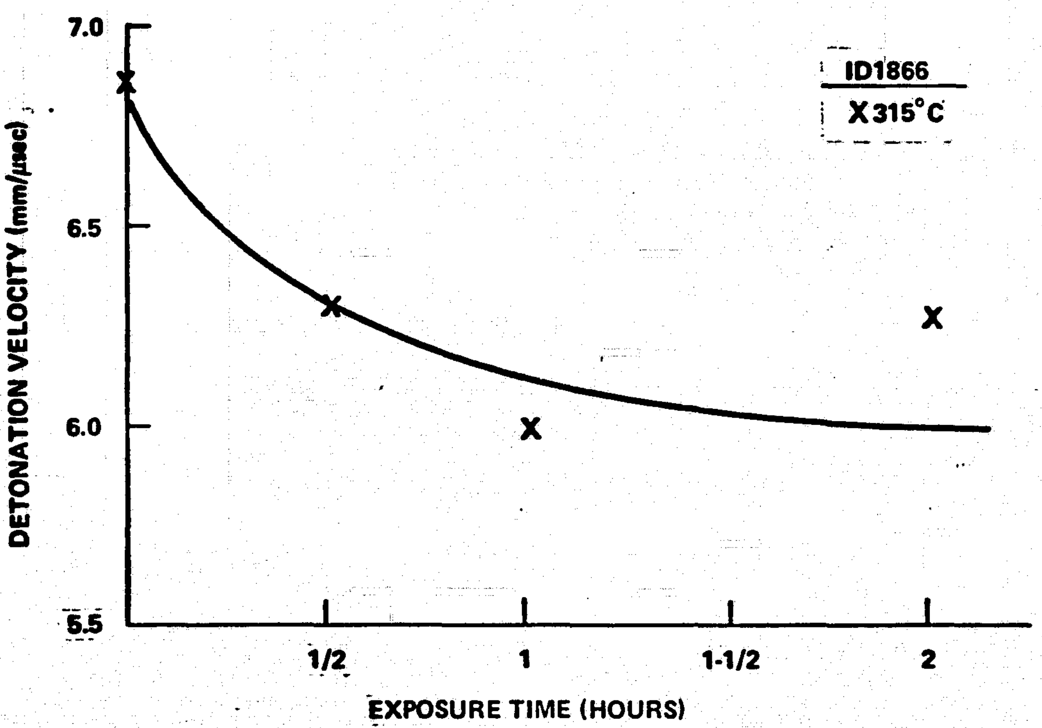


FIG. 12 THE DETONATION VELOCITY OF ONT IN ALUMINUM MDC (0.5g/m OR 2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

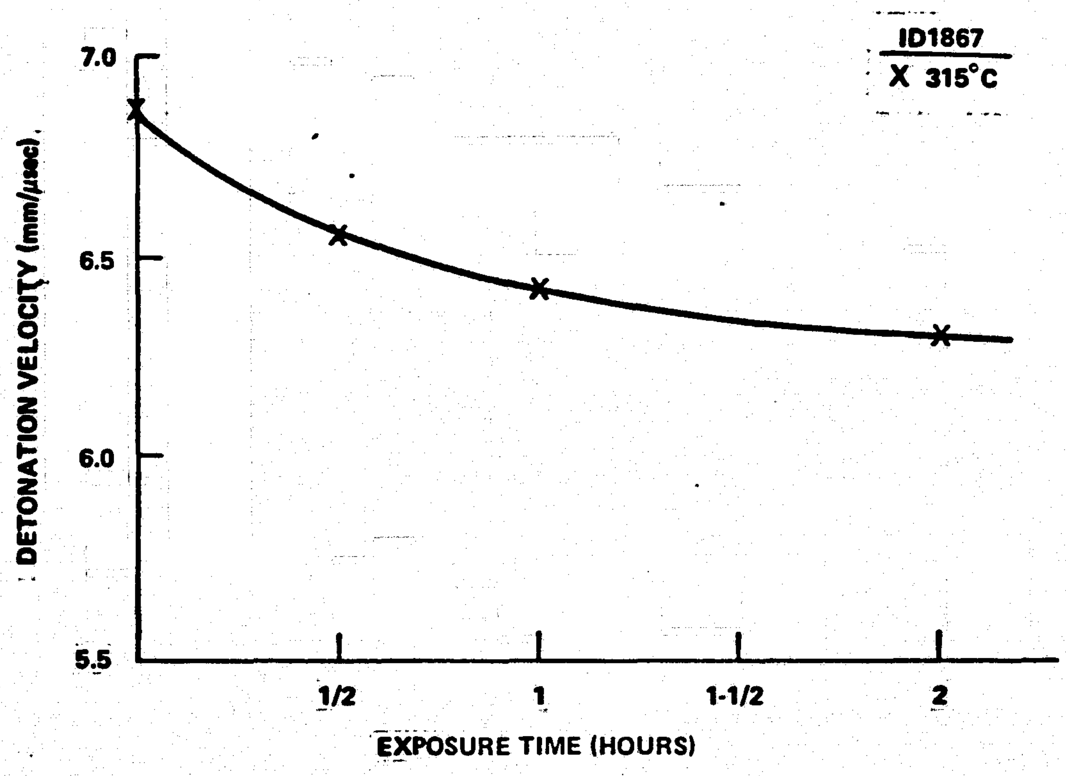


FIG. 13 THE DETONATION VELOCITY OF ONT IN ALUMINUM MDC (2g/m OR 10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

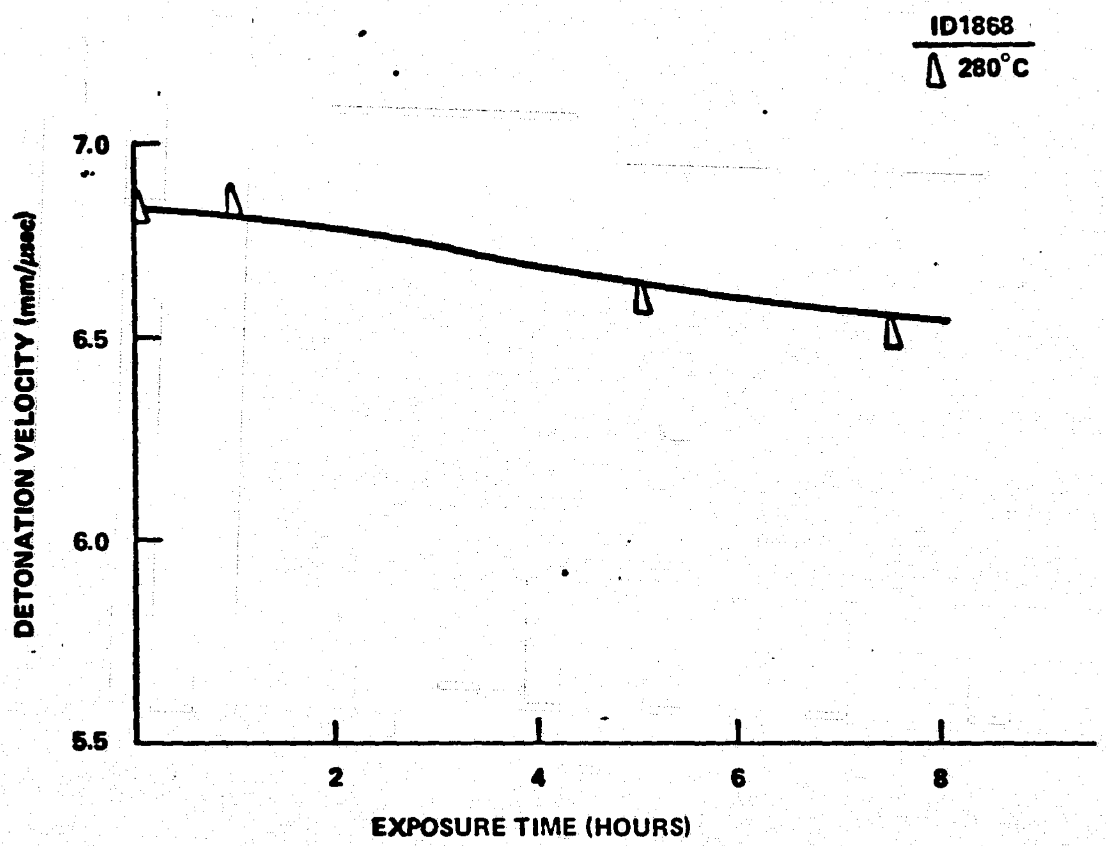


FIG. 14 THE DETONATION VELOCITY OF ONT IN SILVER MDC (0.5g/m OR 2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

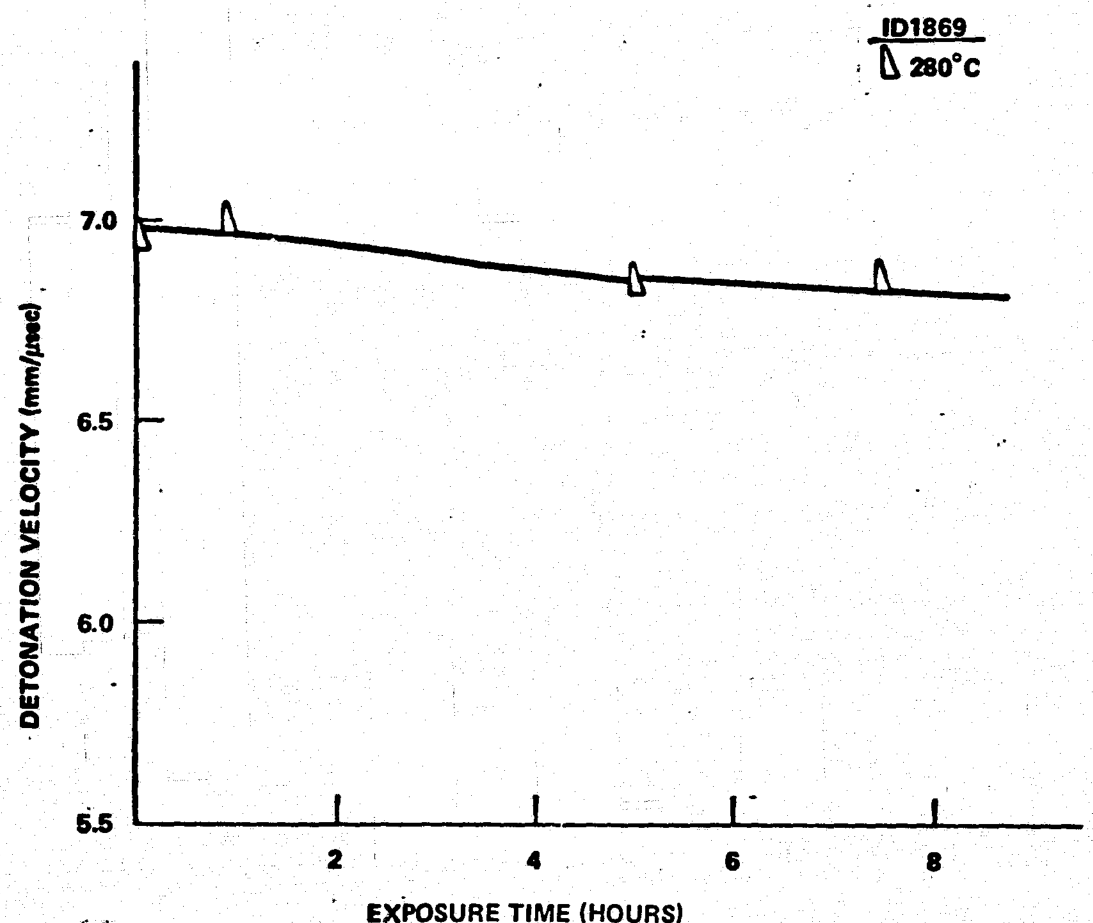


FIG. 15 THE DETONATION VELOCITY OF ONT IN SILVER MDC (2g/m OR 10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

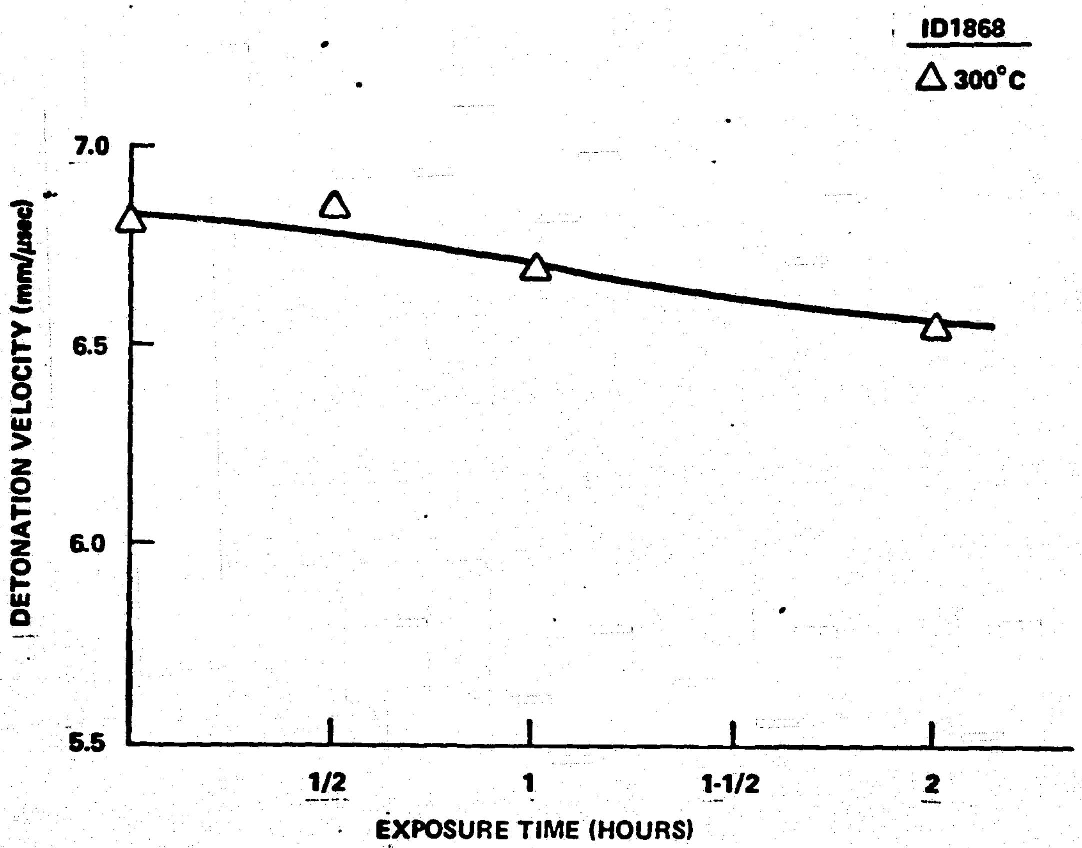


FIG. 16 THE DETONATION VELOCITY OF ONT IN SILVER MDC (0.5g/m OR 2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

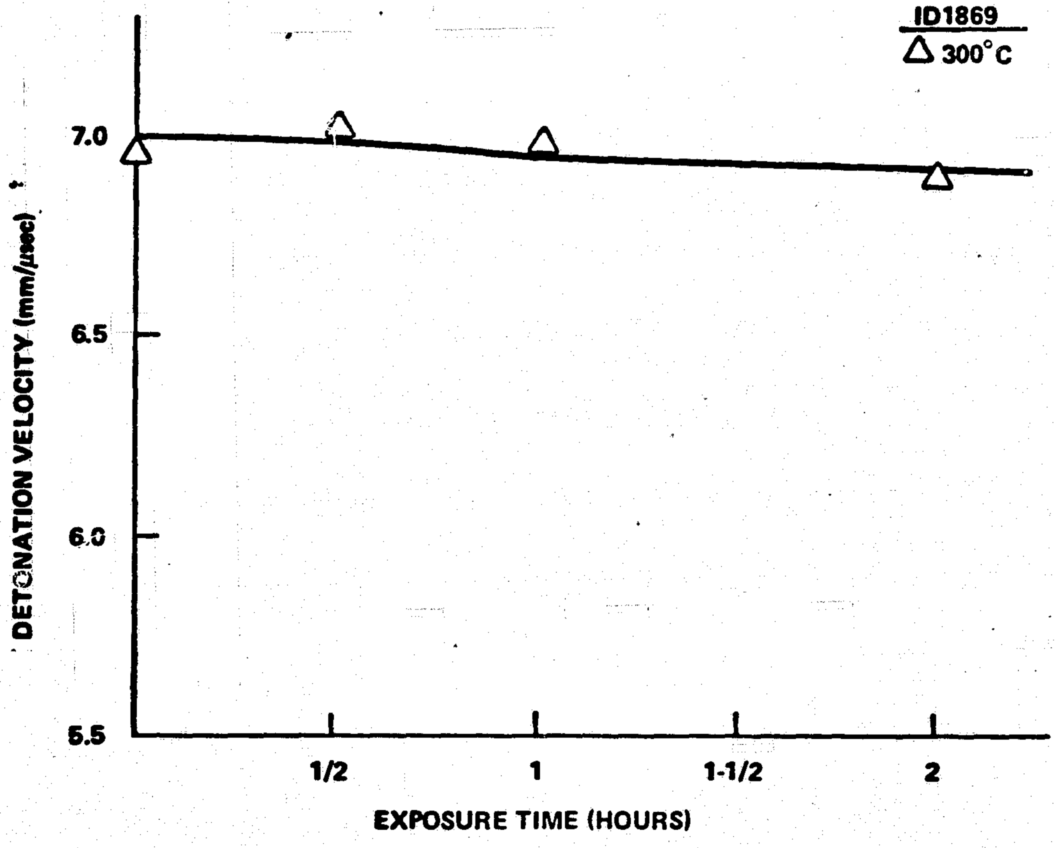


FIG. 17 THE DETONATION VELOCITY OF ONT IN SILVER MDC (2g/m OR 10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

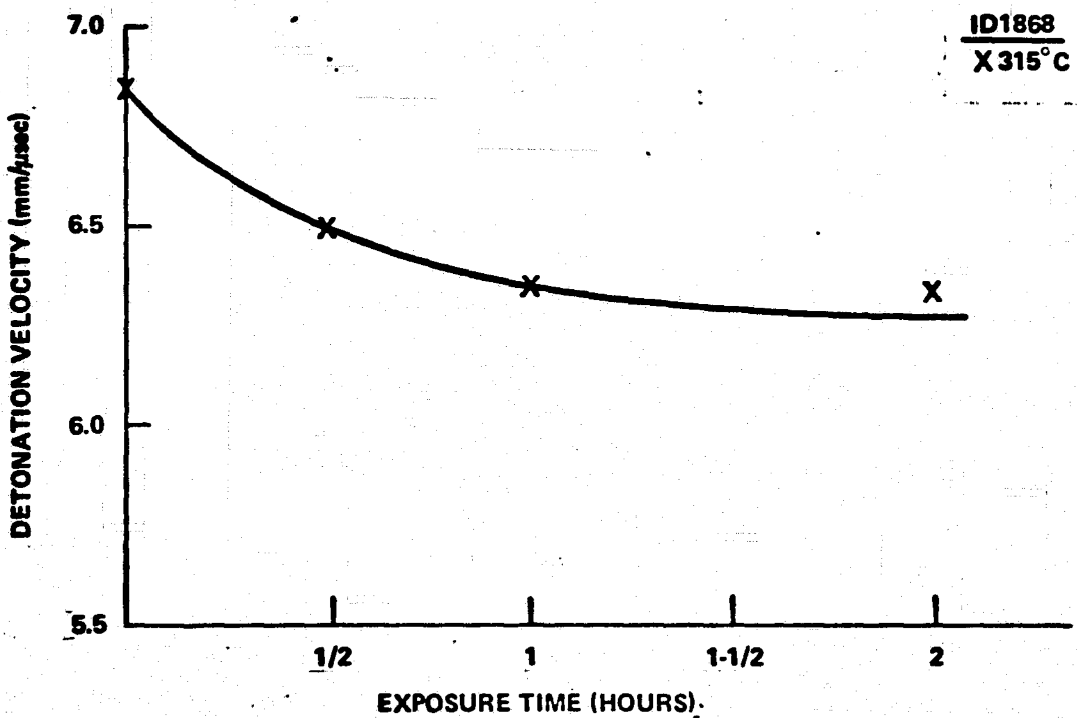


FIG. 18 THE DETONATION VELOCITY OF ONT IN SILVER MDC (0.5g/m OR 2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

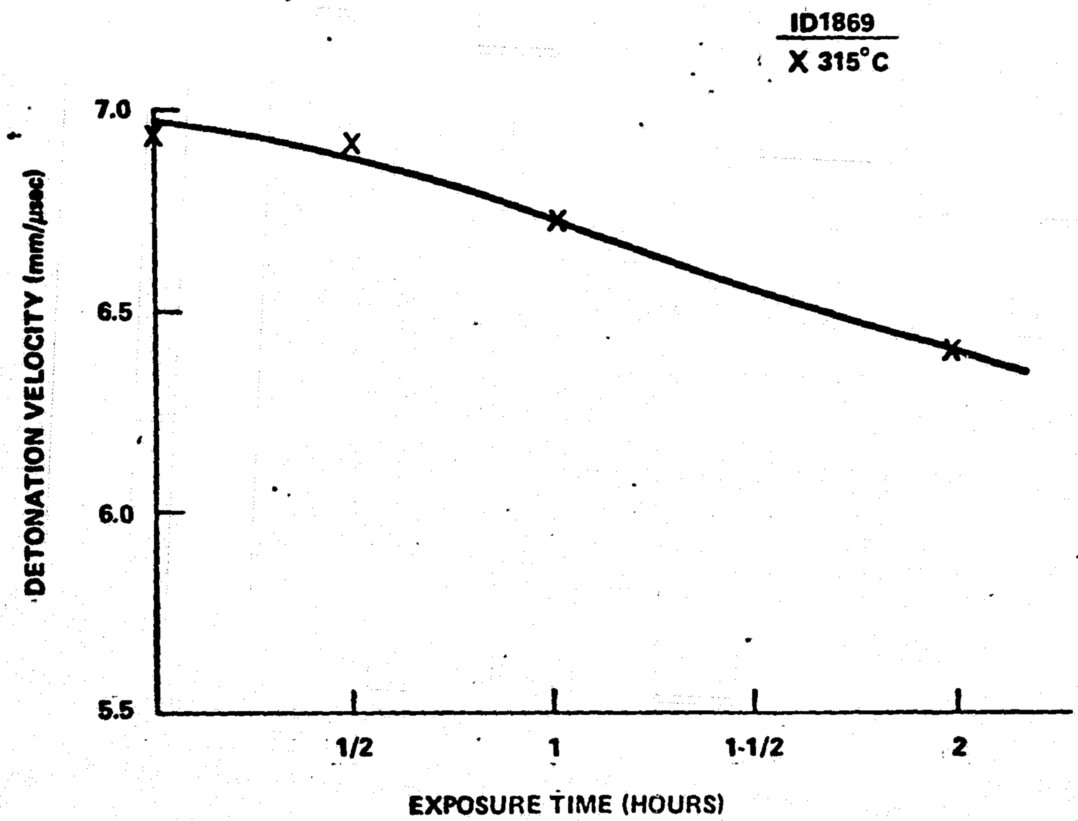


FIG. 19 THE DETONATION VELOCITY ON ONT IN SILVER MDC (2g/m OR 10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

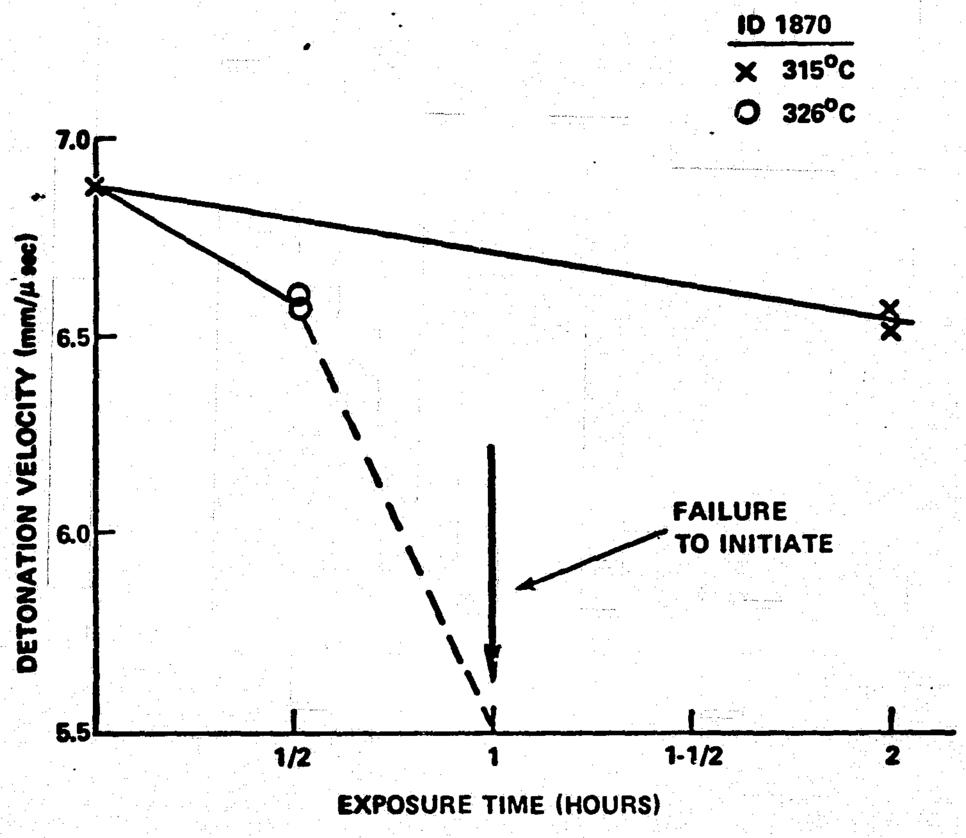


FIG. 20 THE DETONATION VELOCITY OF TPT IN ALUMINUM MDC (0.5g/m OR 2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

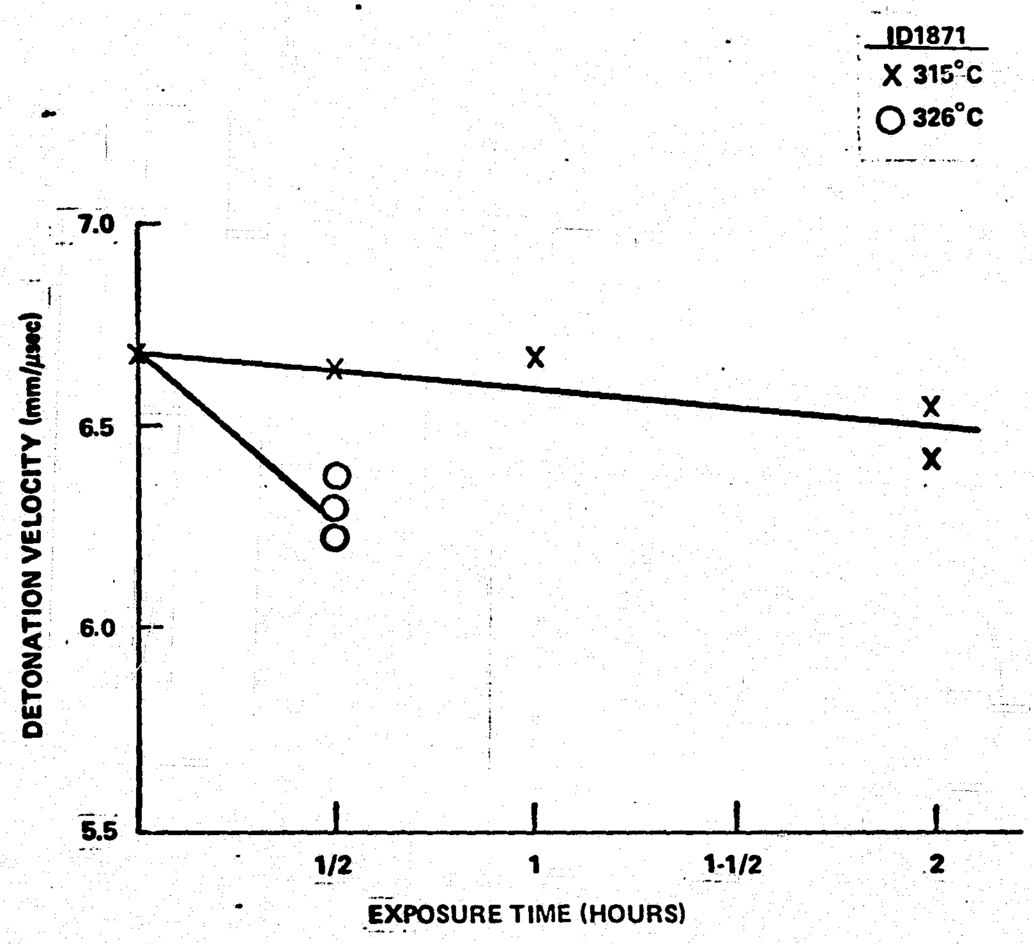


FIG. 21 THE DETONATION VELOCITY OF TPT IN ALUMINUM MDC (2g/m OR 10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

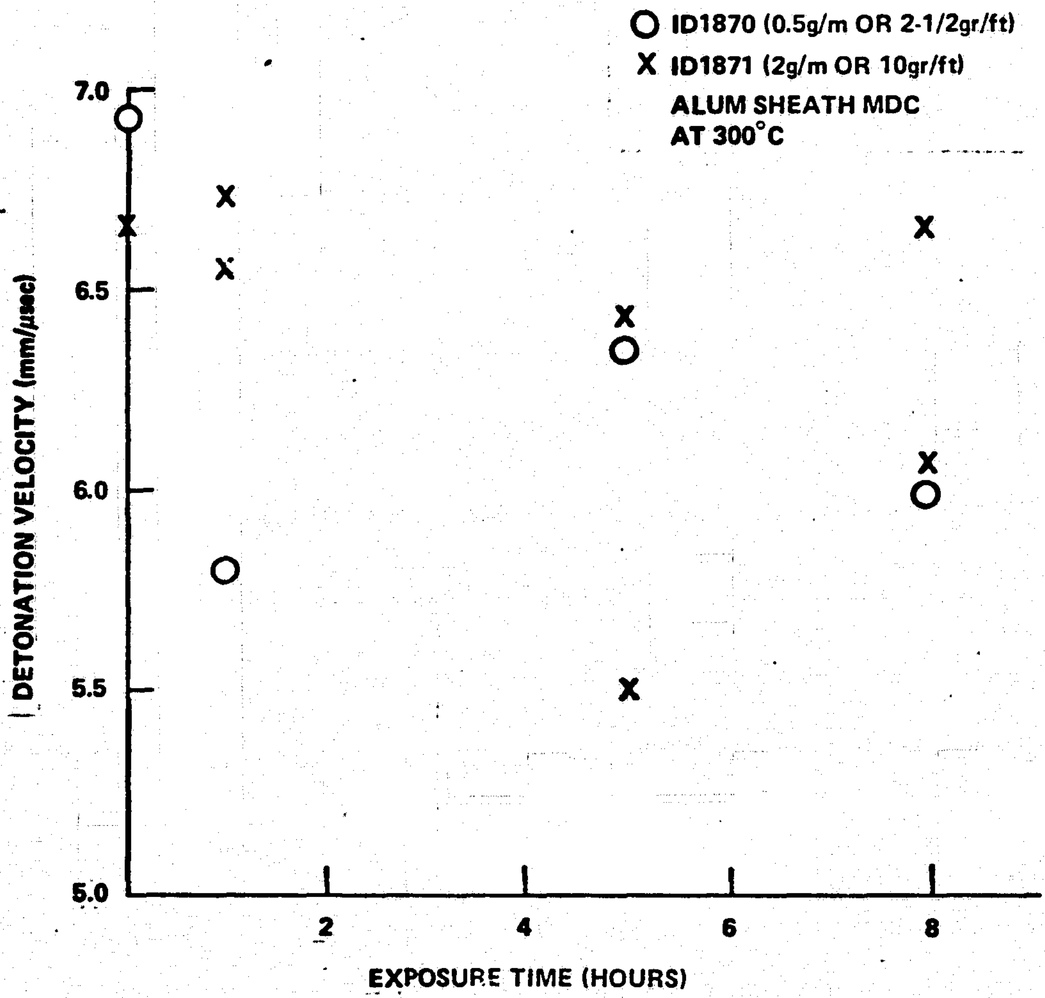


FIG. 22 THE DETONATION VELOCITY OF TPT-MDC (ALUM) AFTER EXPOSURE TO ELEVATED TEMPERATURE

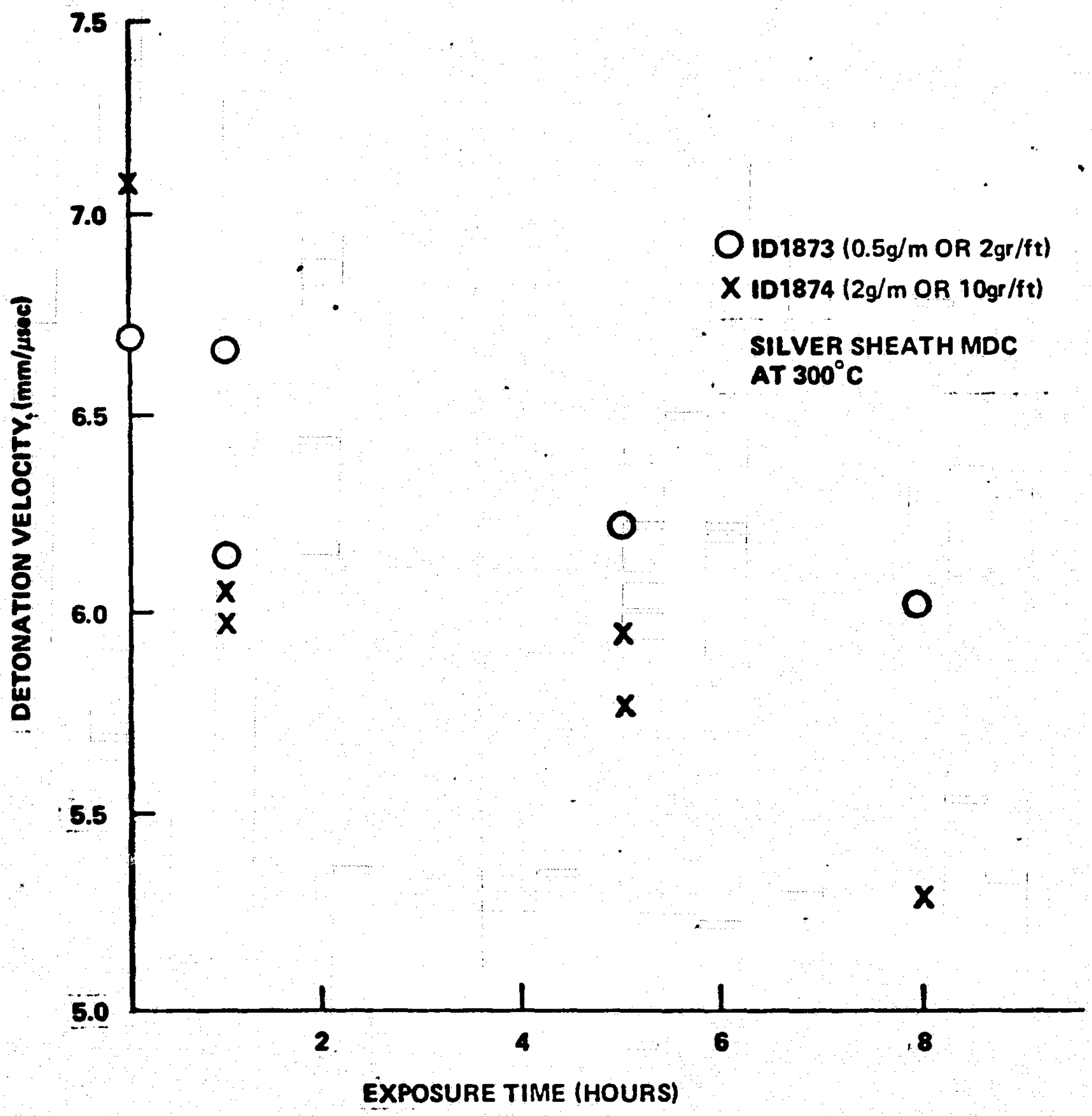


FIG. 23 THE DETONATION VELOCITY OF TPT-MDC (SILVER) AFTER EXPOSURE TO ELEVATED TEMPERATURE

4.3 Even though the statistical sample for the tests was small, the following conclusion is drawn:

TPT has surpassed the design goal for performance after one hour exposure at 600°F. There is little degradation after two hours at this temperature. In addition, it is very likely that this cord will perform after a longer exposure than one-half hour at 326°C (619°F).

5. EXPOSURE OF TPT AND PYX DETONATING CORDS TO ELEVATED TEMPERATURES FOR AN EXTENDED PERIOD OF TIME

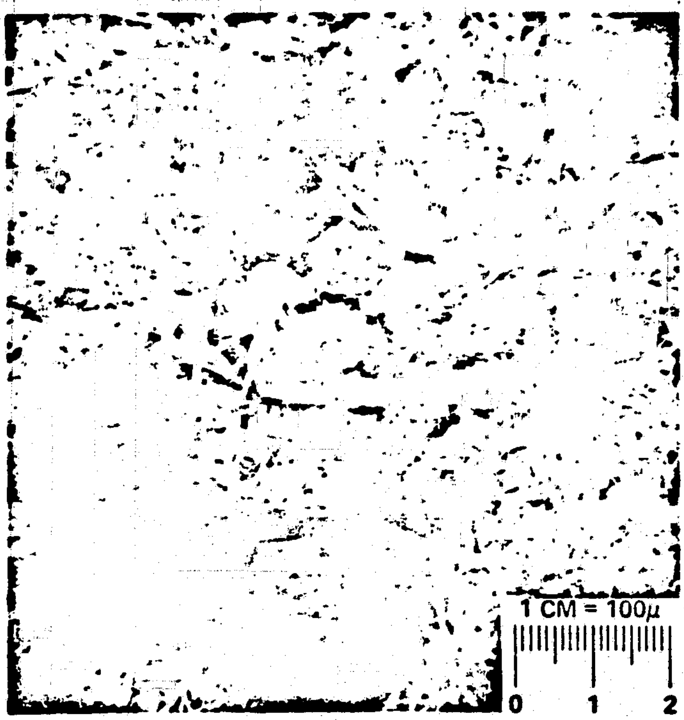
5.1 In the past, NAVSURFWPNCEN developed DIPAM and HNS (Figures 24 and 25) have been exposed to elevated temperatures of 238°C (460°F) for relatively long periods of time[6]. Degradation occupied of such a magnitude as to cause performance problems after about three days at this temperature.

5.2 A better characterization of TPT and PYX explosives could be made if the time of exposure were increased and the temperature decreased. These explosives should show thermal capabilities well above Hexanitrostilbene (HNS), the NAVY's best "in-service" heat resistant explosive, when exposed for periods longer than two hours.

5.3 A study of the performance of these detonating cords was made for periods of time not to exceed 24 hours at temperatures from 260°C (500°F) to 300°C (572°F). The exposures were made in sealed tubes as described above. The results of the detonation velocity measurements are shown plotted in Figures 26 through 33.

5.4 TPT is shown to be more thermally stable than PYX over a 24-hour period of exposure at 260°C in the 2 g/m detonating cords. With the small number of samples tested the reverse appears to be the case for low core load, 0.5 g/m (2.5 gr/ft), detonating cords in silver. The performance of all cords after 280°C exposure (except the 0.5 g/m TPT in silver) appear to be about the same as the functioning after exposure to 260°C. The results are erratic for both materials after exposure to 300°C.

(6) Kilmer, E., "Annual Report on Investigation of High and Low Temperature Resistant Explosive Devices," NOLTR 67-133, 18 Oct 1967



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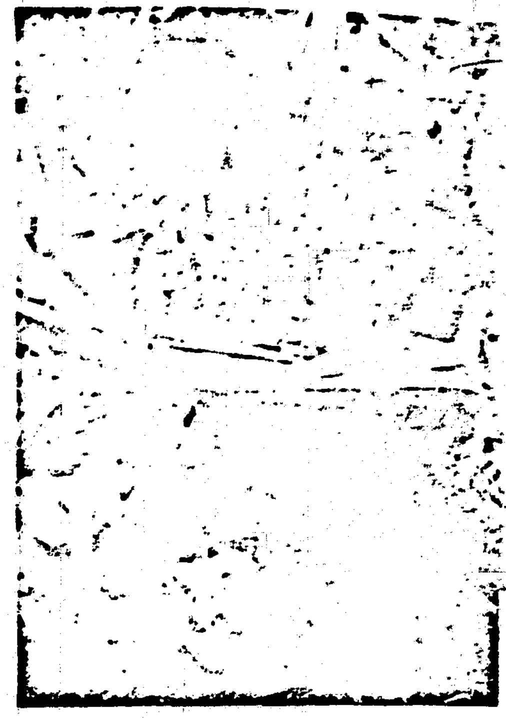

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FIG. 24 SEM PHOTOMICROGRAPHS OF DIPAM (ID203)

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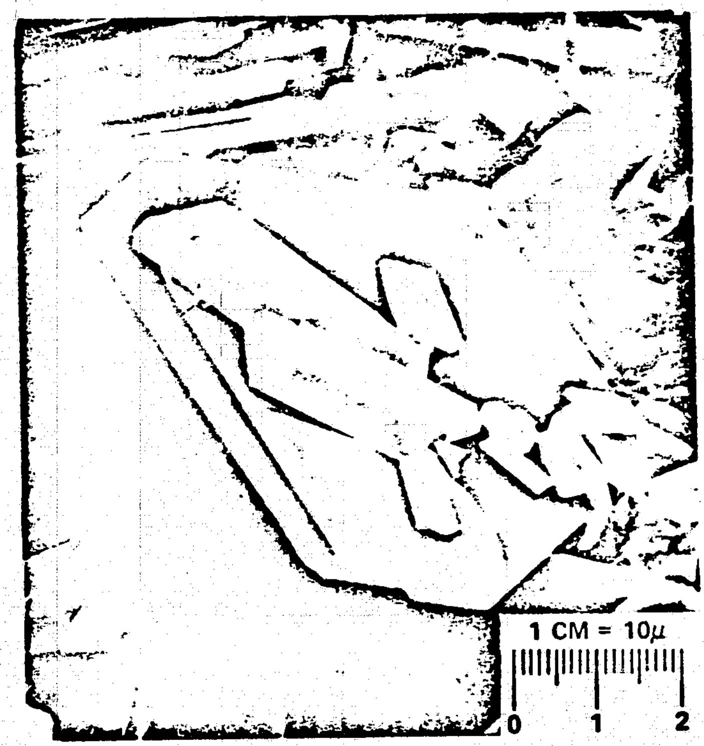

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FIG. 25 SEM PHOTOMICROGRAPH OF HNS-II (ID1479)

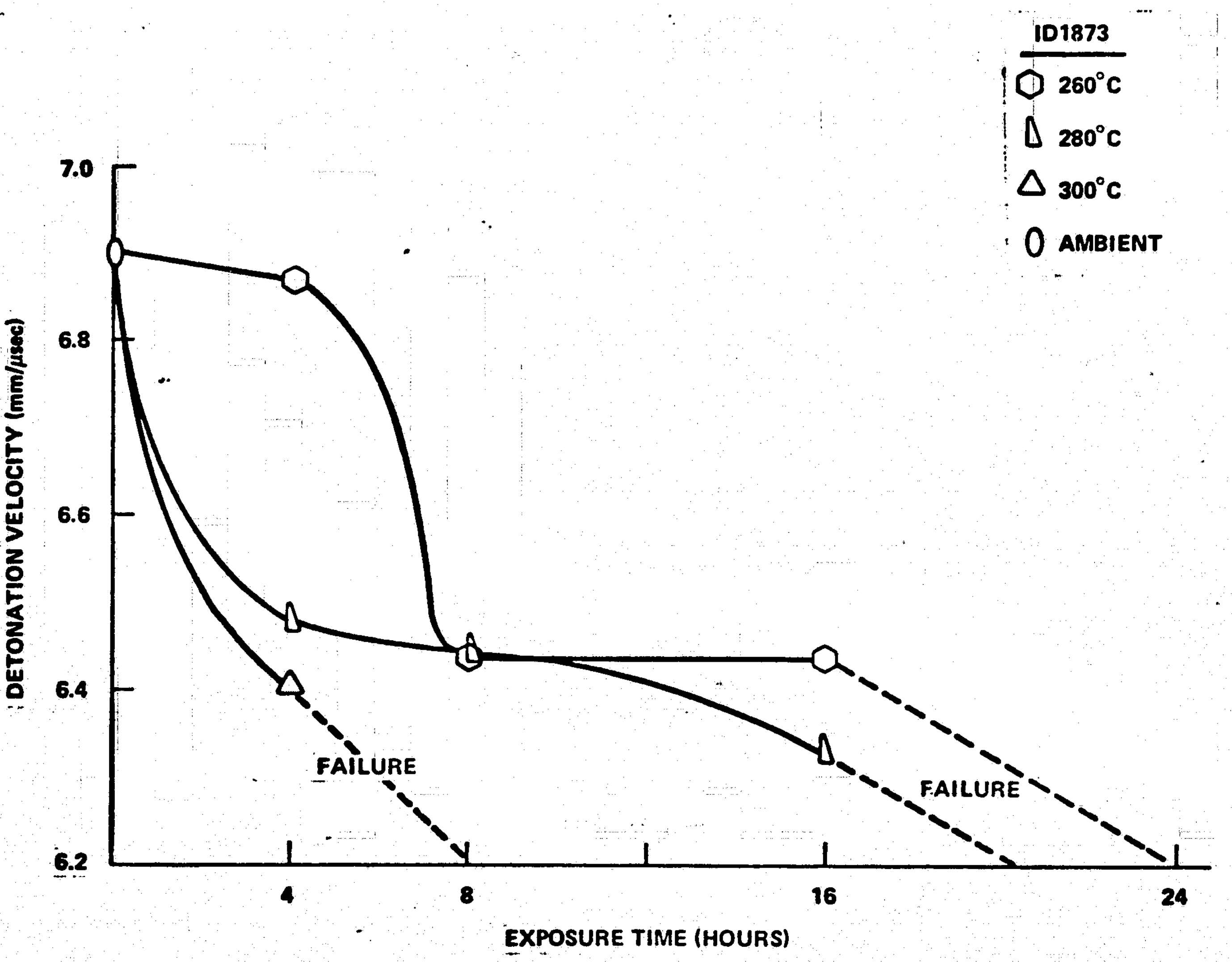


FIG. 26 THE DETONATION VELOCITY OF TPT IN SILVER MDC (0.5g/m OR 2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

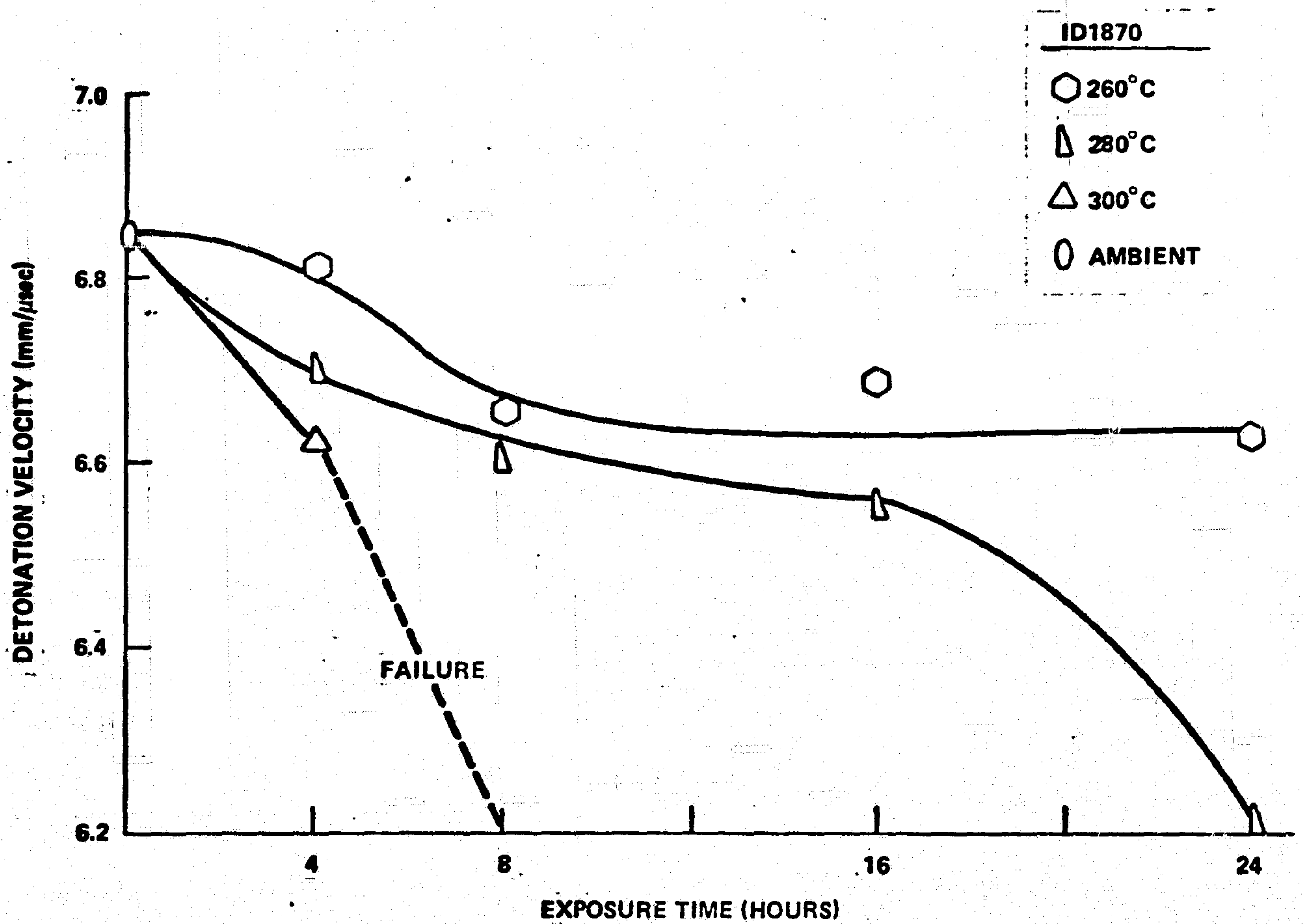


FIG. 27 THE DETONATION VELOCITY OF TPT IN ALUMINUM MDC (0.5g/m OR 2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

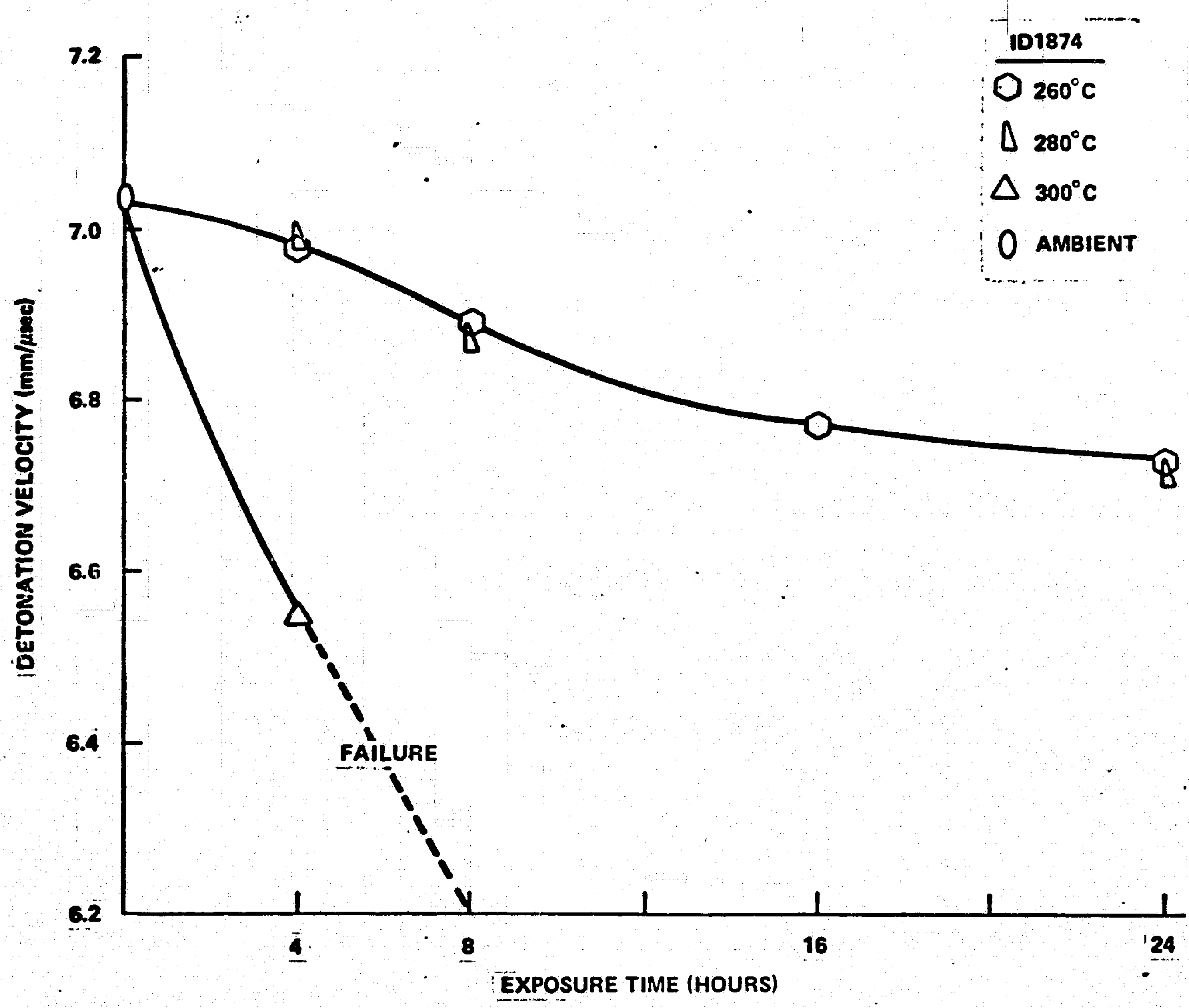


FIG. 28 THE DETONATION VELOCITY OF TPT IN SILVER MDC (2g/m OR 10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

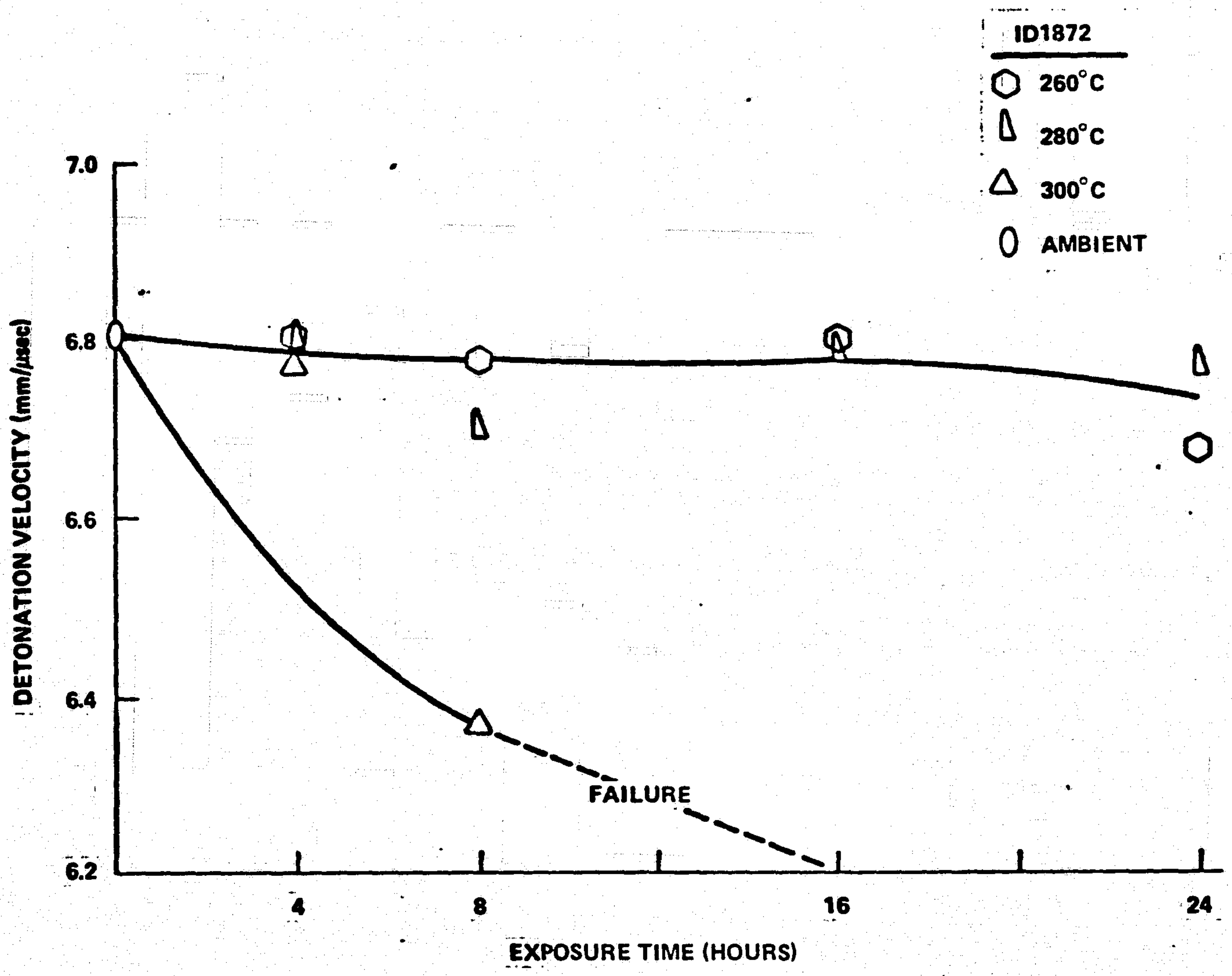


FIG. 29 THE DETONATION VELOCITY OF TPT IN ALUMINUM MDC (2g/m OR 10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

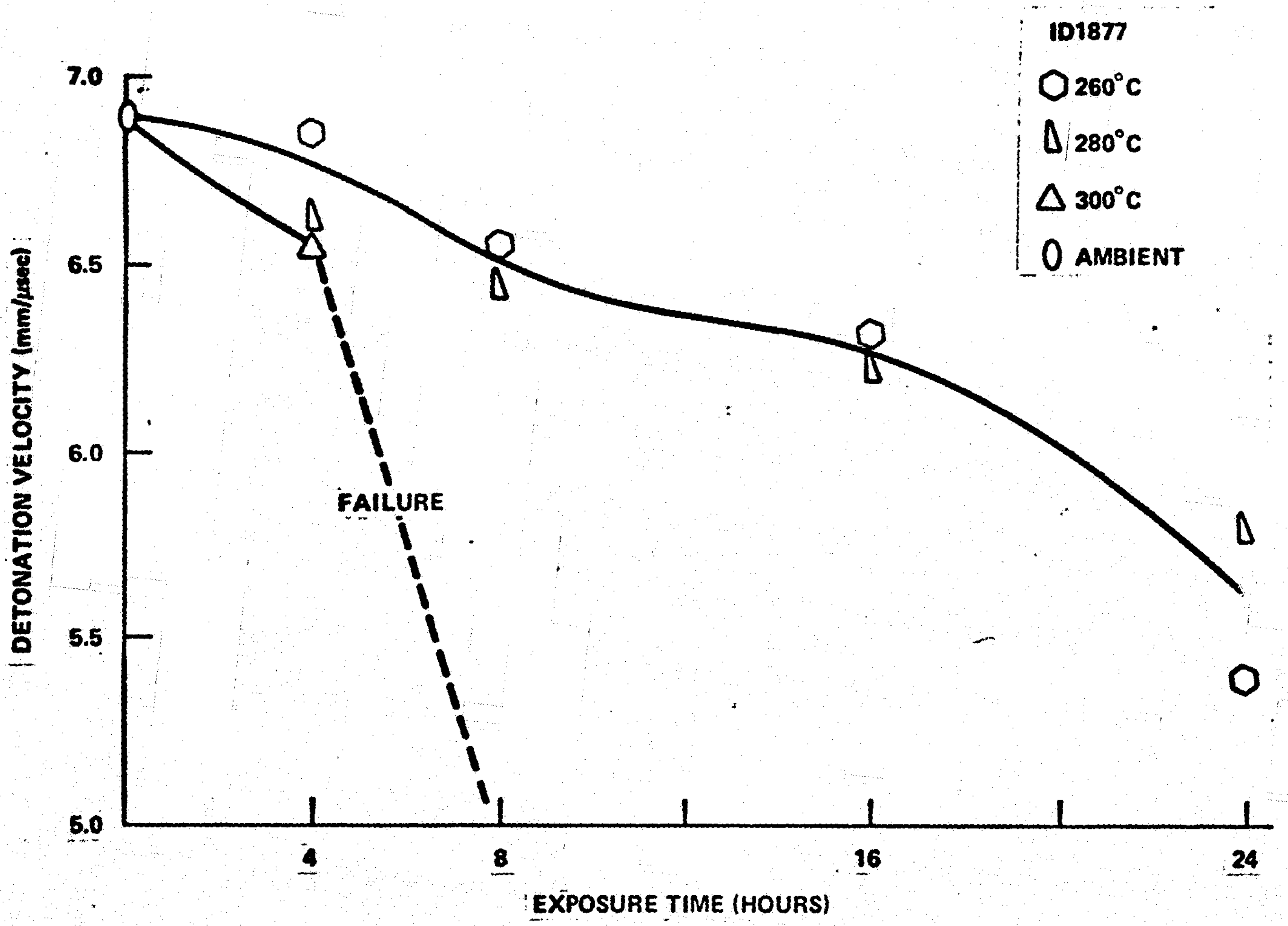


FIG. 30 THE DETONATION VELOCITY OF PYX IN SILVER MDC (0.5g/m OR 2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

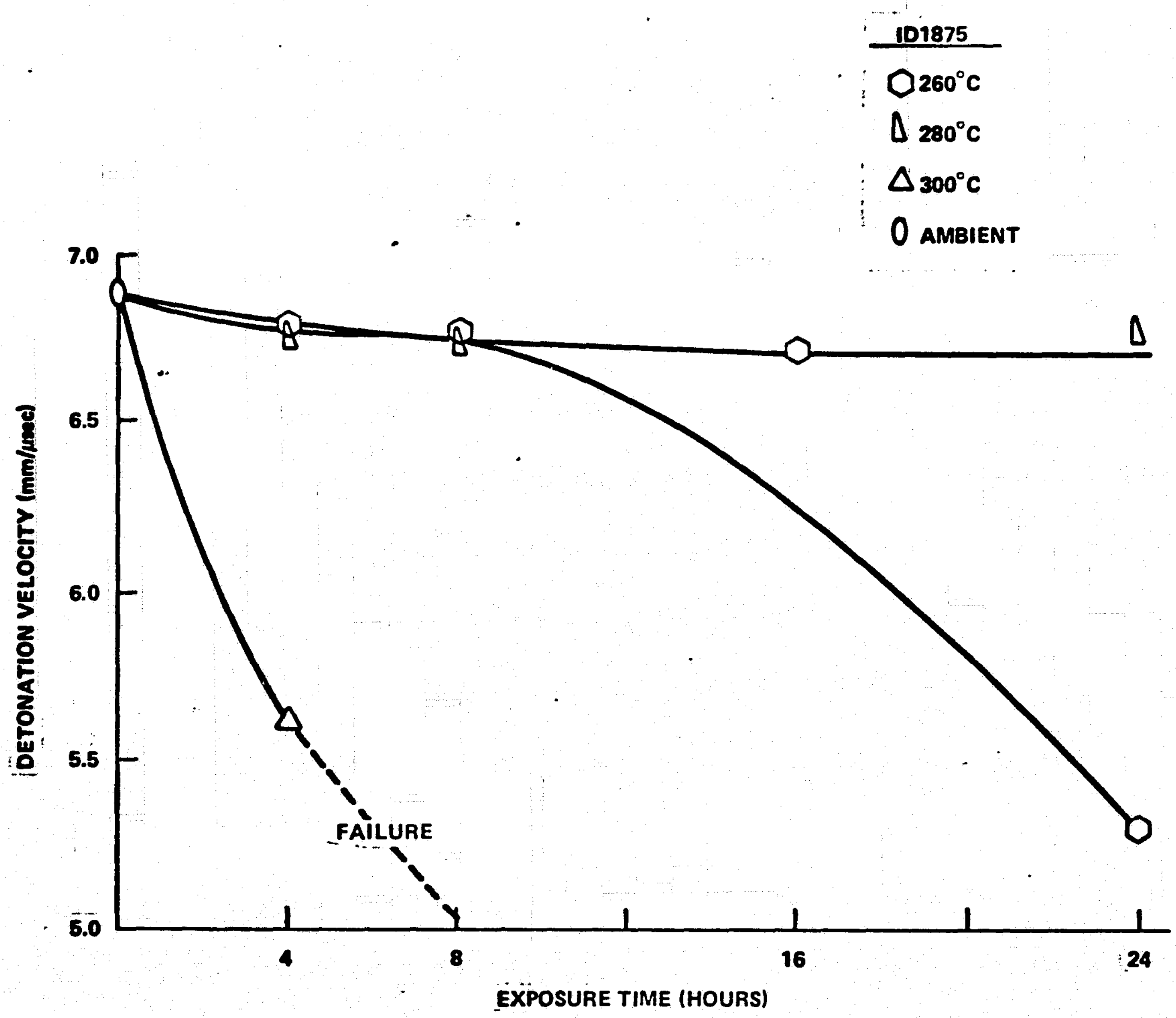


FIG. 31 THE DETONATION VELOCITY OF PYX IN ALUMINUM MDC (0.5g/m OR 2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

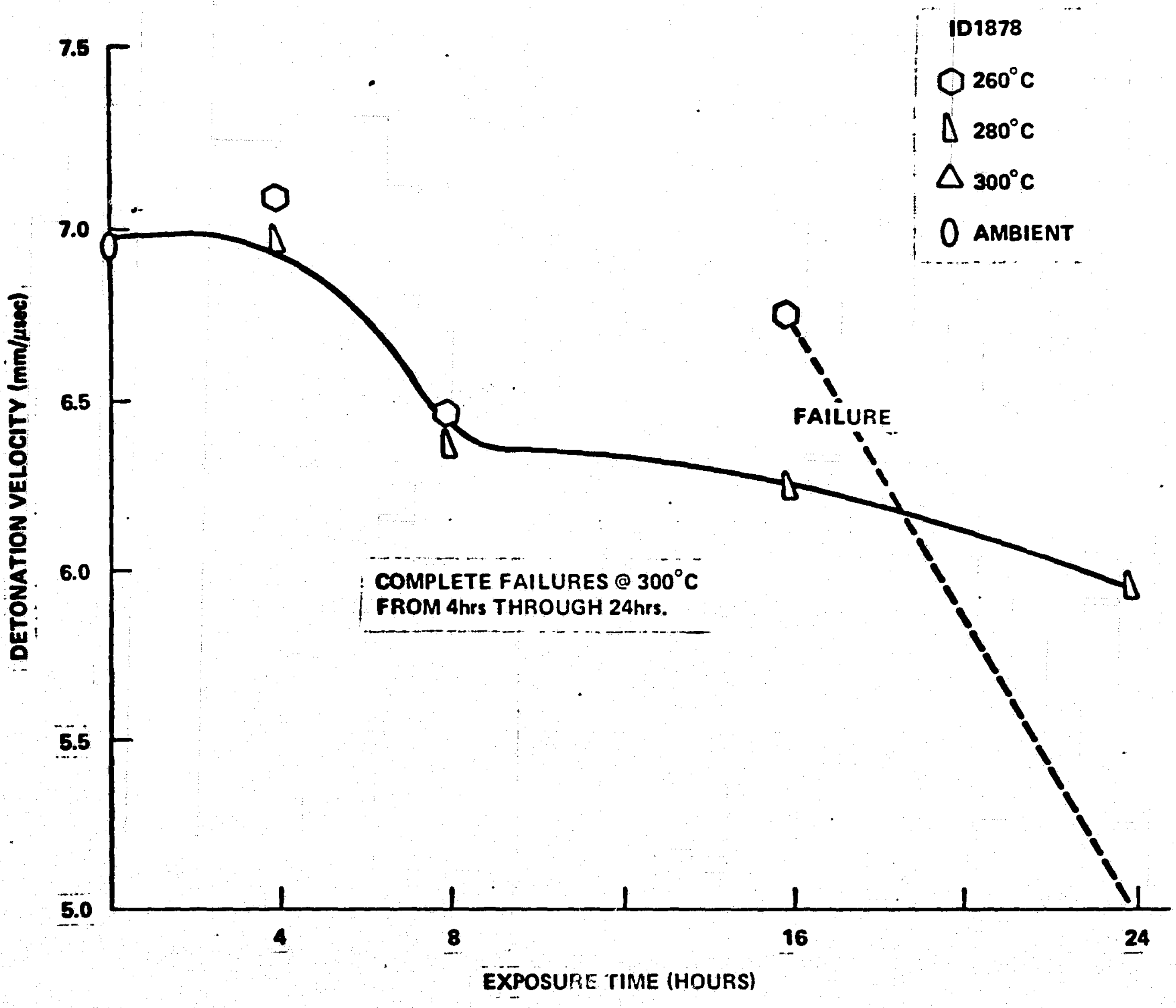


FIG. 32 THE DETONATION VELOCITY OF PYX IN SILVER MDC (2g/m or 10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

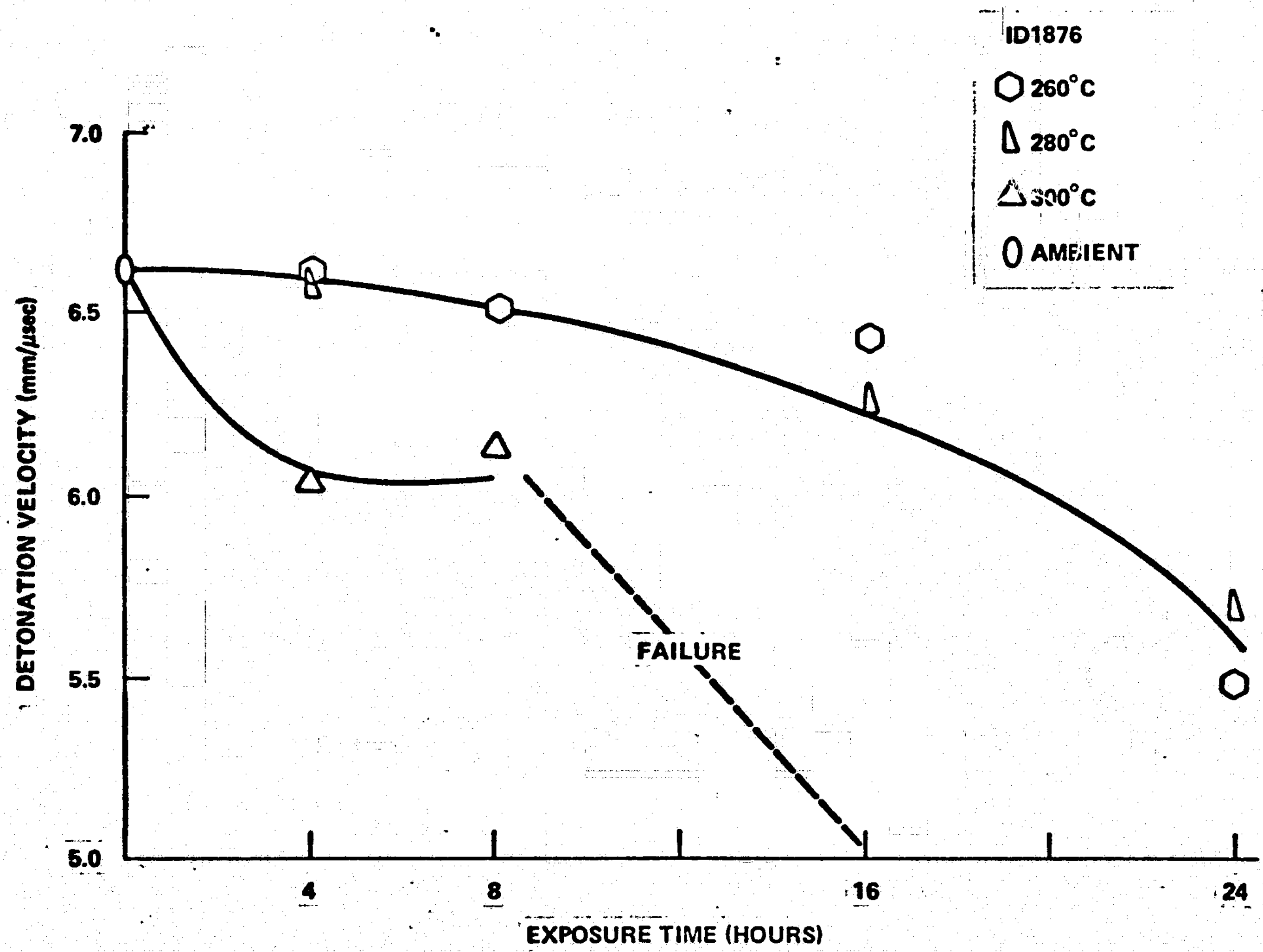


FIG. 33 THE DETONATION VELOCITY OF PYX IN ALUMINUM MDC (2g/m OR 10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

6. CONCLUSIONS/RECOMMENDATIONS

6.1 It has been shown that ONT has surpassed the design goal criterion, but it should be considered to yield marginal performance at 315°C (600°F). ONT does not have as good a thermal stability as PYX and TPT when exposed to very high temperatures for short periods of time in mild detonating cords.

6.2 PYX has been tested in detonating cords and has surpassed the design goal for performance after one hour exposure at 315°C (600°F). There is little degradation after two hours at this temperature in most of the cords tested. If the requirement arises it is recommended that these cords not be exposed for longer than one-half hour at 326°C because of harmful degradation of the explosive especially in the low core load detonating cords.

6.3 TPT detonating cords tested under the same conditions of elevated temperatures as PYX detonating cords performed equally as well for short periods of time at 315°C. When the temperature was raised to 326°C as a penalization test, the results of the performance of the 0.5 g/m PYX cords indicate potential performance problems after 30 minutes of exposures.

6.4 Earlier test results indicated TPT and PYX to be thermally stable for an hour at 315°C. The results of testing after long term exposure indicated poor performance in detonating cords after 4 hours of exposure at 300°C.

6.5 In view of the earlier results[2] on vacuum thermal stability and vapor pressure measurements, TPT was judged to be better than PYX and ONT and was considered to best conform to the NASA guidelines for space use. A material procurement specification NOLS 1022 was written for the TPT explosive.

APPENDIX A

A. Chemical Synthesis of 2,4,6-Tripicryl-s-Triazine, TPT

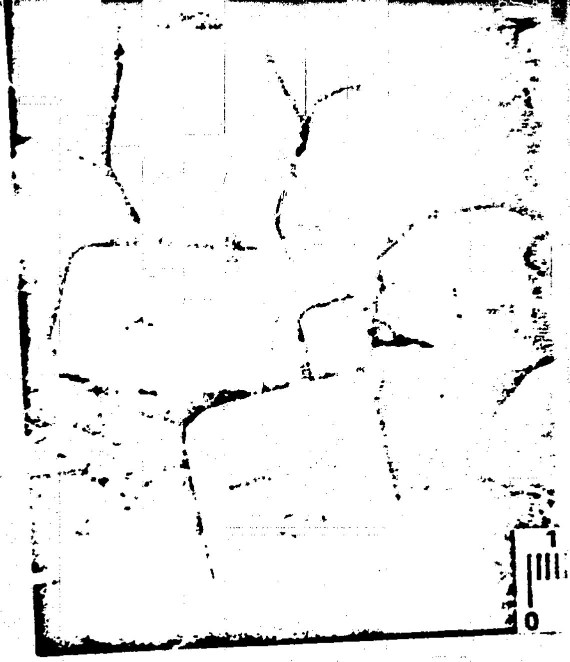
A-1 A study of the chemical synthesis[7] of the thermally stable explosive, tripicryl triazine (TPT), has been made at the NAVSURFWPNCEN by Dr. J. Dacons. The synthesis is documented in the above reference and also by a United States Patent[5]. The original material shown in Figure A.1 was prepared in small quantities by the Ullmann coupling reaction using cyauric chloride in nitrobenzene, powdered copper, and the addition of picryl bromide. This reaction was scaled-up to yield about a 150g quantity of finished product which is three times the batch size of the original preparation. This larger batch size operation was used to prepare six pounds of TPT. Each batch was checked for chemical purity and melting point. This lot of explosive was used to set up the basic requirements for chemical purity and sensitivity for the procurement specification and for the fabrication of detonating cords.

A-2 The chemical synthesis was as follows (reference 7):

Preparation of Picryl Bromide. An amount of 355g (3.5 moles) of reagent grade potassium nitrate was dissolved in 800 ml of 30% oleum in a 2000 ml 3-neck round bottom flask fitted with a mechanical stirrer and a thermometer. During the addition of the potassium nitrate, the mixture was cooled and stirred on an ice bath, the temperature being kept below 60°C. When the addition was complete, the mixture was cooled at 30°C and 79g (0.5 moles) of bromobenzene was added at such a rate as to keep the temperature below 50°C. The ice bath was then replaced by an oil bath and the reaction mixture was heated to approximately 125°C for four hours. After cooling, the mixture was drowned in crushed ice, the product was collected by filtration (using a sintered glass funnel) and thoroughly washed with water. After drying in a warm oven, it was dissolved in 100 ml of acetone. 300 ml of methanol was added and the solution was cooled in the freeze compartment of the refrigerator. On filtration, 83.7g of pale yellow crystalline picryl bromide, mp 120-122°C (lit. 122-3°C) was recovered. The filtrate was concentrated to about 150 ml by boiling on the steam bath and again cooled in the freeze compartment. The second crop of precipitate weighed 28.0g and melted over the range 111-118°C. On recrystallization from acetone-ethanol, an additional 21.0g of crystalline product, mp 120-122°C was obtained to give a total yield melting at this temperature of 104.7g, 71.6%. Samples melting at 122-123°C were obtained by one additional recrystallization from acetone-methanol; however, the product melting at 120-122°C was of sufficient purity for use as a cord explosive.

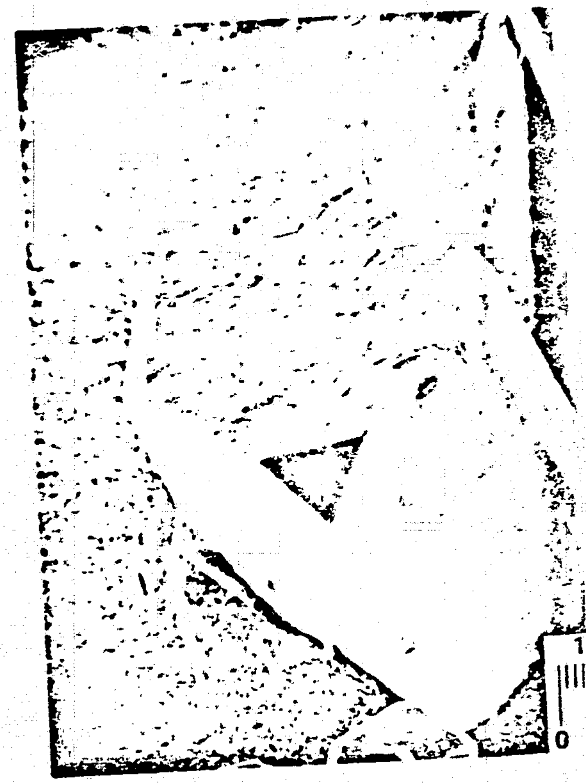
(7) Dacons, J. C. and Sitzmann, M. E., "Heat Resistant Explosives XXVI. The Synthesis and Properties of 2,4,6-tripicryl-s-triazine, TPT," NOLTR 68-64, 29 May 1968

100 X **ID 1103**



250 X **ID 1103**

500 X **ID 1103**



400 X **ID 1103**

FIG. A-1 SEM PHOTOMICROGRAPHS OF TPT (ID 1103) NSWC ORIGINAL PREP.

A-3 Preparation of 2,4,6-Tripicryl-s-triazine, TPT. A solution of 18.5g (0.1 moles) of cyanuric chloride in 100 ml of dry nitrobenzene was made in a 500 ml 3-neck round bottom flask fitted with a mechanical stirrer, an addition funnel, an air cooled condenser and a thermometer. To this solution was added 89.0g (1.4 moles) of copper powder* and, with stirring, the temperature was raised to 140°C on an oil bath held in place by a laboratory jack. A volume of 20 ml of a solution of 188g (0.4 moles) of picryl bromide in 150 ml of dry nitrobenzene was added rapidly. The temperature dropped to 134°C and there was an immediate reaction indicated by a change in the color of the copper and a rise in the temperature of the mixture to 146°C. Heating was reduced by lowering the oil bath and dropwise addition of the remainder of the picryl bromide solution was started. The rate of addition was adjusted so as to require approximately 40 minutes for its completion. A temperature range of 140-146°C was maintained during the addition and for 20 minutes thereafter. The reaction mixture was allowed to cool to ambient temperature and then poured into 700 ml of methanol and cooled to 5°C. It was then filtered with suction through a glass fiber filter and the residue was thoroughly washed with cold methanol. After drying in a warm oven, the residue containing the TPT along with cuprous halides and unused copper, was digested in 1000 ml of approximately 20% hydrochloric acid with stirring for about 20 minutes, then filtered through sintered glass. The residue, which was mainly TPT and copper, was washed first with dilute hydrochloric acid, then with water and finally with methanol until the washings were nearly colorless. After drying on the funnel for a few minutes, the filter cake was heated in three liters of acetone to dissolve the TPT, treated with 25 g of Darco G 60 and filtered. The amber filtrate was concentrated by rapid distillation with stirring until considerable crystallization had occurred and the volume was about 400 ml. Methanol was then added at about the same rate as the distillate was removed until the temperature of the distillate was 62°C. The mixture was then cooled to 5°C on an ice bath, the product was removed by filtration, washed with cold methanol and dried in a warm oven. The yield was 60.7 g of a fine, very pale yellow crystalline solid which melted at 350-1°C (d) after sintering at approximately 140°C.

A-4 In order to obtain high purity TPT, the above material was dissolved in 2000 ml of acetone and recrystallized as described above. On drying in a warm oven overnight, the product was 55.5g of glistening pale yellow crystals, mp 352-3°C (d) after sintering at 140°C. The product was a solvent which on drying overnight at 140°C under reduced pressure lost its solvent of crystallization leaving 50.4g (72% yield) of porous opaque crystals, mp 352-3°C (d).

*Purified Copper Powder from Matheson, Coleman and Bell and Venus Natural Copper Fine No. 44-F, U. S. Bronze Powders, Inc., Flemington, New Jersey have been found suitable for this reaction.

A-5 In order to determine the nature of the solvate, a twice recrystallized sample, which had been dried at 80°C was heated at 280°C for thirty minutes and the gas evolved was vacuum transferred to a gas chromatograph. The retention time identified the gas as acetone. Samples which had been dried overnight at 140°C showed no acetone content. By measuring the weight loss on heating at 140°C for extended periods, it was found that the solvate contained 7.5% acetone and 92.5% TPT corresponding to one mole of acetone per mole of TPT. (See Figure A.2).

A-6 A crystal density determination was made of the acetone solvate and was found[8] to be 1.64g/cc as shown in Figure A.3. This value is low because of the nature of the recrystallization. The material was recrystallized from dichlorobenzene to improve the crystal density to 1.72g/cc which is the best value for the unsolvated crystal (Figure A.4).

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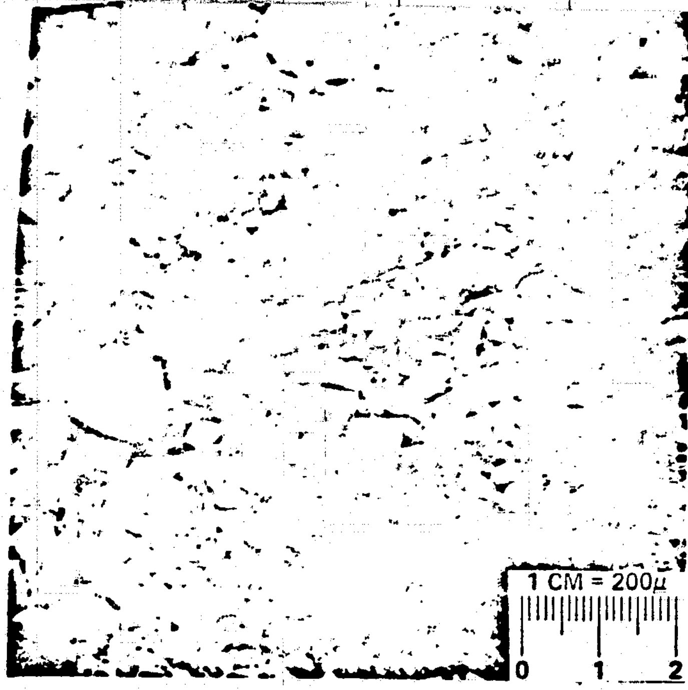
(8) Dacons, J., "2,4,6-Tripicryl-s-Triazine, TPT: It's Crystallization and Crystal Density Determination," NSWC/WOL/TR 76-16, 8 Mar 1976, Unclassified



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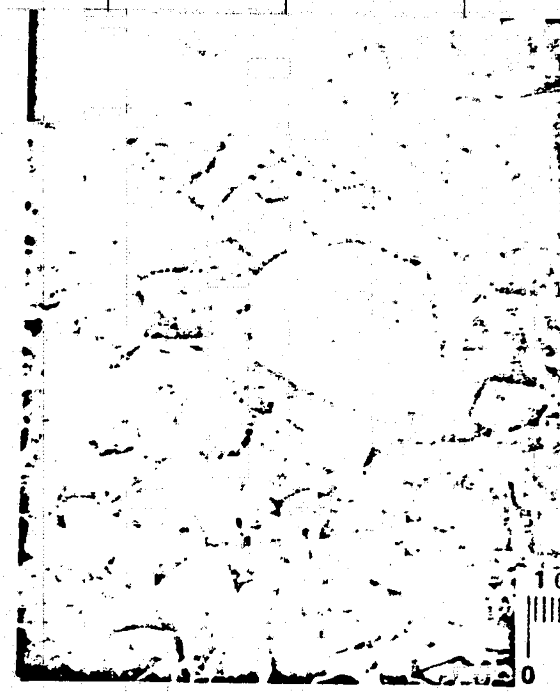

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500 X ID 1888

FIG. A-2 SEM PHOTOMICROGRAPHS OF TPT (ID1888) PREPARED AT NSWC FOR SPECIFICATION

30 X ID 2063

100 X ID 2063

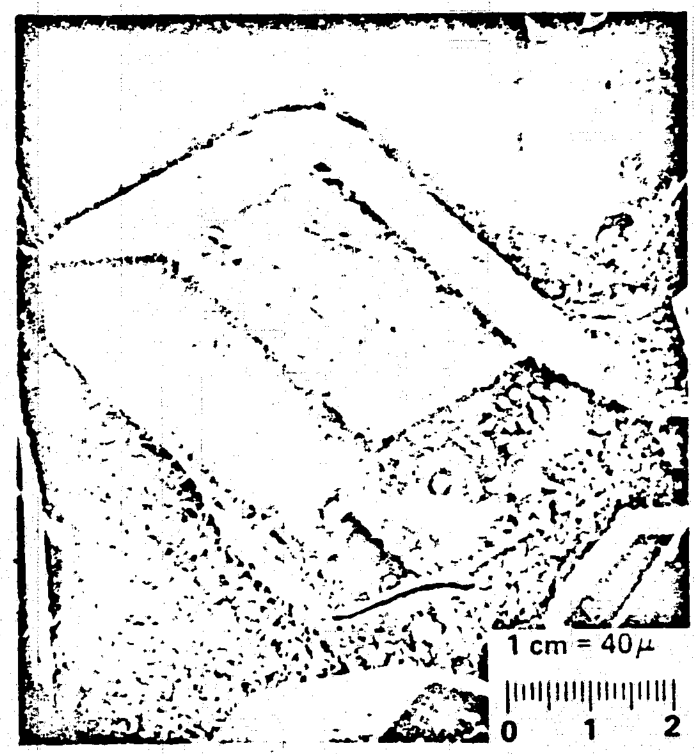

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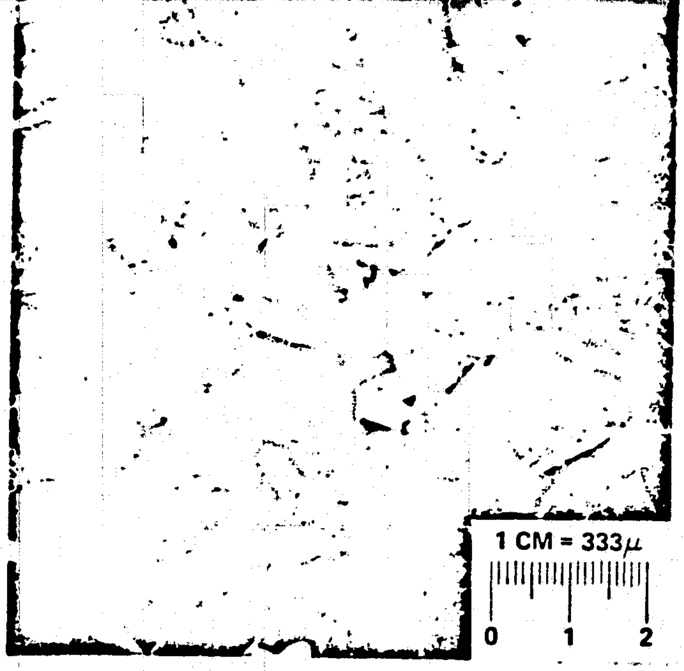
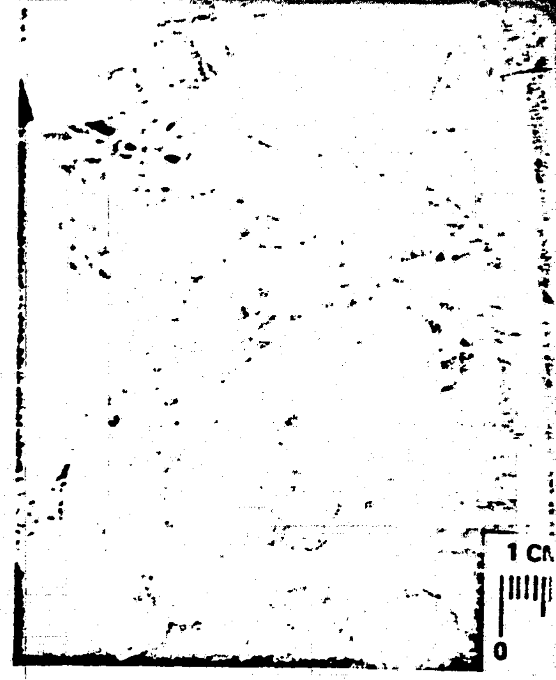
FIG. A-3 SEM PHOTOMICROGRAPHS OF TPT (ID2063) SOLVATED CRYSTAL ρ=1.64g/c



30 X ID 2065



100 X ID 2065

250 X ID 2065

FIG. A-4 SEM PHOTOMICROGRAPHS OF TPT (ID2065) UNSOLVATED CRYSTAL ρ=1.72g/c

APPENDIX B

B. The Dynamic Compressibility of TPT

B-1 It is desirable in the characterization of any high explosive to measure accurately its shock sensitivity. The impact sensitivity test and the small scale gap test do not give refined values of shock pressure required to determine when the explosive will be initiated to deflagration or detonation. One measure of the sensitivity of an explosive to shock is the pressure needed to cause a chemical reaction. This information is available from measurements of the dynamic compressibility of the unreacted explosive. The pressure needed to cause reaction is important in designing shock initiated explosive components and in providing protection against accidental explosions.

B-2 The dynamic pressure-volume curve (shock Hugoniot) of TPT was determining from experiments which measured shock wave velocities in TPT samples and the shock properties of calibrated driver plate materials. Four pressure-volume points and two sound velocity points were measured in the TPT samples*.

B-3 The experimental arrangement used to determine the shock Hugoniot is shown in Figure B.1. A plane wave explosive lens shocks a driver plate on which the explosive samples are attached. The shock wave arrivals are recorded by a smear camera using a reflected light technique[9, 10]. In this experimental technique light is reflected continuously from the free surfaces into the camera. The shock wave arrival at any point along the reflective surfaces produces a sudden change in light reflected from that point. These changes in intensity are recorded on the camera film as a function of time. Measurements on the film give the time required for the shock wave to travel through the test samples. This data along with the thicknesses of the test samples allows the velocity of the shock wave to be calculated. The free-surface velocity of the driver plate material also is measured using the reflected-light technique.

*(A small amount (5%) of TEFLON was added to allow the TPT to be pelletized.)

(9) Coleburn, N. L., J. Chem. Phys., 40, 71, (1964)

(10) DuVall, G. E. and Fowles, G. R., "Shock Waves," High Pressure Physics, Vol. 2, pp 209, Academic Press (1963)

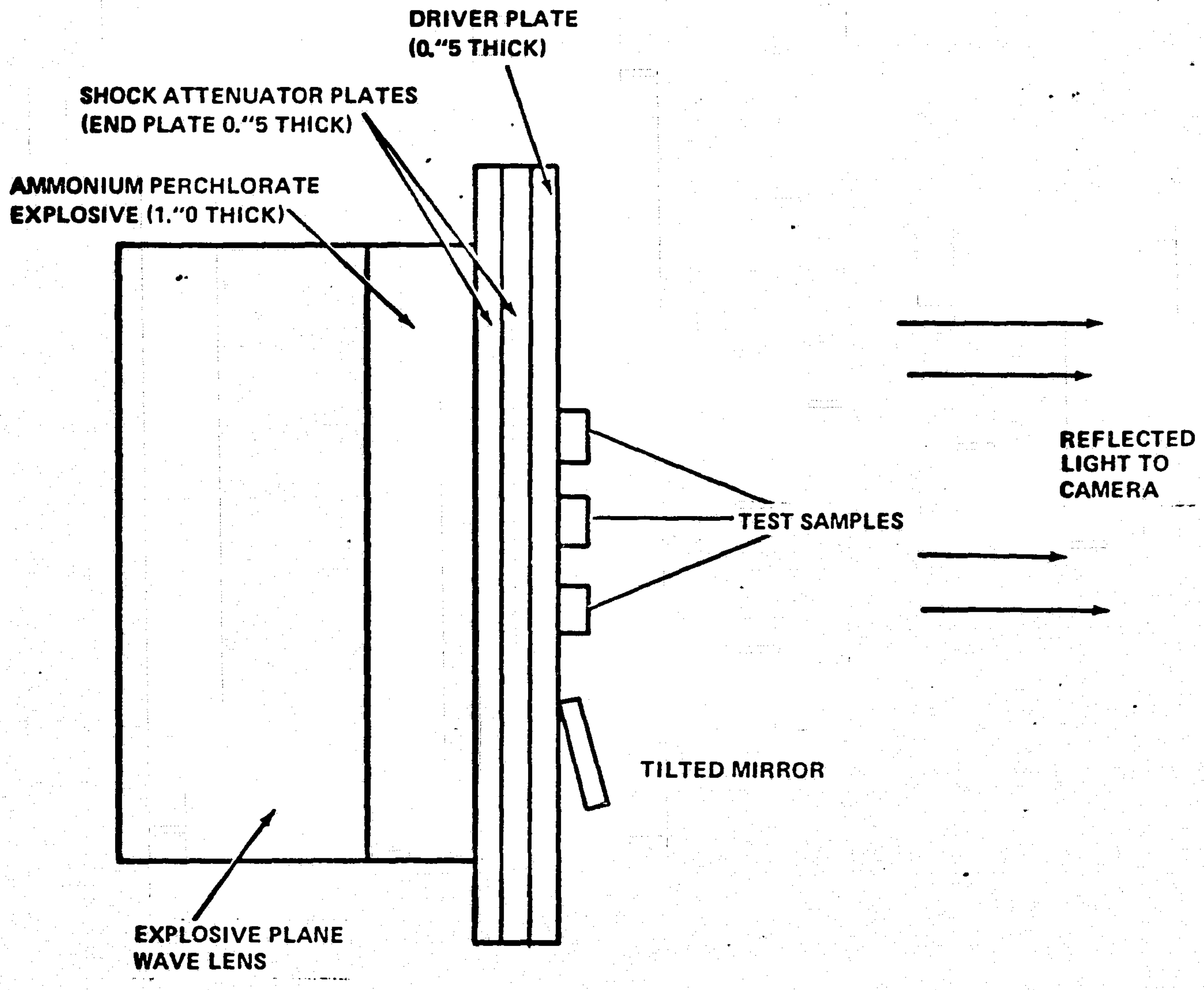


FIG. B-1 EXPERIMENTAL SETUP (SIDE VIEW)

B-4 The measured shock wave parameters were used in the conservation laws to obtain the pressure, volume, and particle velocity in the shocked materials. The conservation laws[11] are:

$$P = \rho_o U_s u_p \tag{1}$$

$$\frac{V_1}{V_0} = \frac{U_s - u_p}{U_s} \tag{2}$$

where P is the pressure behind the shock front, ρ_o is the initial density, U_s is the shock velocity, u_p is the particle velocity behind the shock front. (u_p = 1/2 the free-surface velocity.) (In using these conservation laws the assumptions of steady flow and no chemical reaction were made.)

B-5 It is obvious from equation (1) that the measurement of shock velocity and particle velocity are sufficient for determining the pressure in the driver plate. This pressure and the measured shock wave velocity in TPT was used in an impedance-matching data reduction technique[11] to determine the state parameters (i.e., P, V, u_p).

B-6 The experimental results of pressure, shock velocity, and particle velocity obtained so far for TPT are summarized in Table B-1. The shock velocity-particle velocity data is represented by a straight line.

$$U_s = 1.35 \text{ mm/microsec} + 2.56\, u_p \text{ mm/microsec.} \tag{3}$$

When chemical reaction occurs, a large deviation from linearity in a plot of the data will result. The data indicate that no significant reaction occurs at pressures up to 38 kilobars. Pressure necessary for reaction to begin will be established by additional experiments.

B-7 A bulk sound velocity of 1.34 mm/microsec was estimated in TPT using a weak shock technique[12,13] to measure the velocities of longitudinal elastic waves in TPT at pressures of about 100 bars. (The density of the test samples was 1.5 g/cm^3). This value of the bulk sound speed is in excellent agreement with the intercept (u_p = 0) of the U_s, u_p curve as it should be in accord with shock theory.

(11) Duvall, G. E., "Shock Waves in Condensed Media," Physics of High Energy Density, pp 41, Academic Press (1971)
(12) Coleburn, N. L., and Liddiard, T. P. Jr., J. Chem. Phys., 44, 1929, (1966)
(13) Coleburn, N. L., J. Acoust. Soc., 47, 269, (1970)

TABLE B-1
EXPERIMENTAL RESULTS OF SHOCK COMPRESSIBILITY TESTS

EXPERIMENT NUMBER	SHOCK ATTENUATOR PLATES*	DRIVER PLATE**	PRESSURE IN DRIVER PLATE (KBAR)	TPT			
				INITIAL DENSITY (GM/CM^3)	SHOCK VELOCITY (mm/μ sec)	PARTICLE VELOCITY (mm/μ sec)	PRESSURE (KBAR)
1	ALUMINUM/ PLEXIGLAS	ALUMINUM	83	1.57	3.21	0.75	38
2	BRASS/ POLYETHYLENE	ALUMINUM	73	1.52	3.15	0.68	32
3	BRASS/ POLYETHYLENE	BRASS	82	1.58	2.40	0.42	16
4	BRASS/ PLEXIGLAS	BRASS	80	1.61	2.30	0.38	14

*SHOCK SYSTEM WERE 6.25–INCH DIAMETER PLANE WAVE BOOSTERS WITH 1.0–INCH THICK AMMONIUM PERCHLORATE POD AND TWO 0.5–INCH THICK SHOCK ATTENUATOR PLATES.

**DRIVER PLATES WERE 0.5–INCHES THICK.

B-8 The dynamic pressure-volume curve for TPT is compared with the curve for TNT[13] in Figure B.2. Note that at any specific pressure TPT has a smaller volume ratio, V/V_o than TNT. Therefore, TPT is more compressible than TNT.

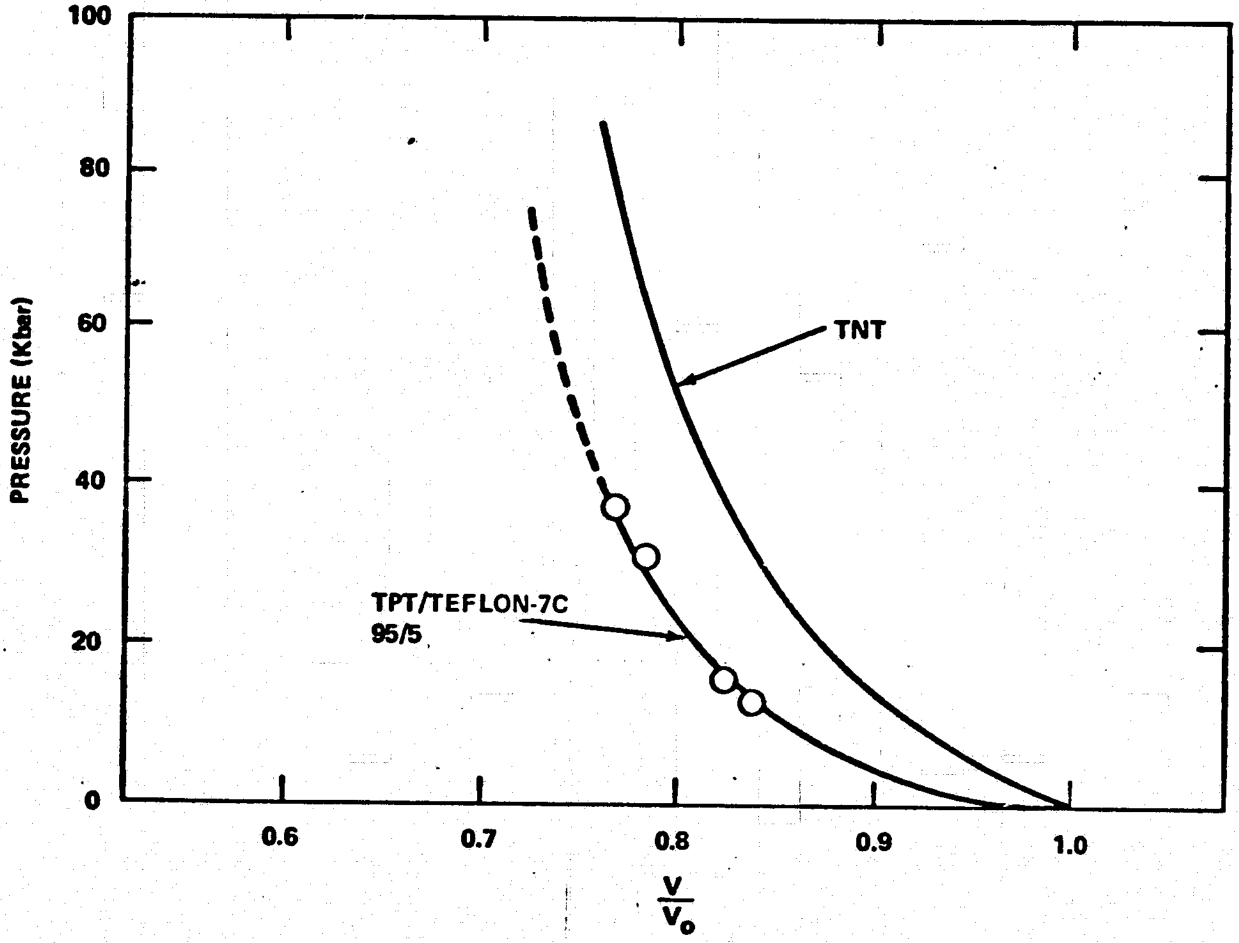


FIG. B-2 SHOCK COMPRESSIBILITY CURVE FOR UNREACTED TPT AND TNT

C. Determination of Heat of Formation of TPT

C-1 As a part of the characterization studies for these heat resistant explosives, the heats of combustion of TPT was determined through the use of a rotating-bomb calorimeter. The heat of formation of TPT was determined by Baroody and Carpenter of the Indian Head Facility of the Naval Surface Weapons Center. A mixture of the explosive and diethyl oxalate (DEO) was used for the combustion process. The results, along with the crystal density of TPT, can be used to obtain estimates of detonation velocity and pressure and heat of detonation.

C-2 The enthalpy of formation of 2,4,6-tripicryltriazine (TPT) was determined by combustion calorimetry, using a platinum-lined rotating-bomb calorimeter. A mixture of TPT and diethyloxalate (DEO) was used to desensitize and ensure complete combustion of TPT. The enthalpy of formation (298.15k) was determined from the following idealized equation at one atmosphere:

$$C_{21}H_6O_{1i}N_{12}(c) + 13.5\ O_2(g) \rightarrow 21\ CO_2\ (g) + 3H_2O\ (l) + 6N_2\ (g)$$

C-3 Several trial runs were conducted on TPT with several solvents in a Parr stationary adiabatic-bomb calorimeter to determine the heat auxiliary material to use with TPT. Also, the proper weight ratios to approximate the heat released for initial temperature settings on the rotating bomb, and to test for ignitability and good combustion of TPT mixtures.

C-4 The enthalpy of formation of TPT was determined to be:

$$\Delta H_f^{\circ} = 7.56 + 0.99 \text{ k cal/mol}$$